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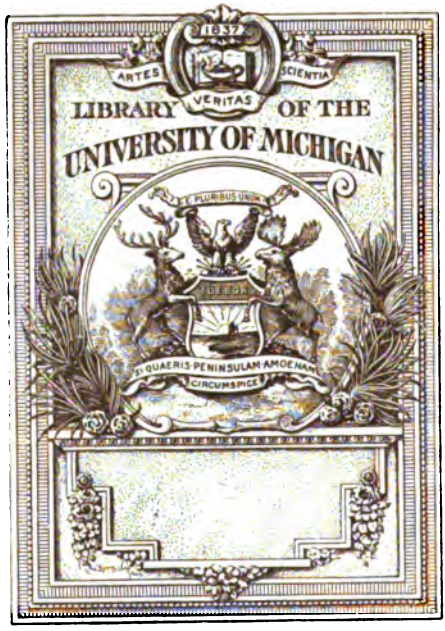
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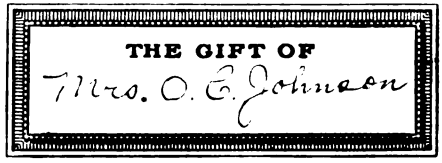
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INORGANIC

GENERAL, MEDICAL AND PHARMACEUTICAL

CHEMISTRY

THEORETICAL AND PRACTICAL

A TEXT-BOOK AND LABORATORY MANUAL

CONTAINING

THEORETICAL, DESCRIPTIVE, AND TECHNOLOGICAL CHEMISTRY; CLASS EXERCISES IN CHEMICAL EQUATIONS AND MATHEMATICS; AND PRACTICAL MANUFACTURING PROCESSES FOR FIVE HUNDRED CHEMICAL PREPARATIONS, WITH EXPLANATORY NOTES

BY

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IN TWO VOLUMES

VOLUME II.—ILLUSTRATED

1900

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PREFACE TO THE SECOND VOLUME.

The laws and conditions which govern chemical reactions and their direction, velocity and relative approach to completion have been treated of in the first volume, including the necessary conditions of success in preparation work so far as they may be indicated by general principles. The materials and methods employed for the production of inorganic pharmaceutical preparations were pointed out in a general way, the subject of oxidation and reduction was fully discussed, and the use of chemical equations and stœchiometry explained and exemplified.

Part I of the second volume discusses more fully the intelligent choice of methods, materials and apparatus, and the practical manipulations of actual laboratory operations in the production of inorganic preparations, and Part II contains detailed descriptions of the modes of preparation of five hundred inorganic chemicals. These processes should be of practical value to pharmacists and manufacturing chemists as well as to teachers and students. Chemical laboratory work in the schools has in the past been almost exclusively analytical work; but the at least equal value and importance of practical work in the production of chemical compounds is now fully recognized.

OSCAR OLDBERG.

School of Pharmacy of Northwestern University,
Chicago, 1900.

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PART I.

GENERAL PRINCIPLES AND METHODS.

CHEMICAL PREPARATIONS.

CHAPTER I.

THE CHOICE OF METHODS AND MATERIALS.

1. The methods of preparation by which inorganic pharmaceutical products are obtained may be physical processes not resulting in any alteration of the molecules of the materials operated upon, or they may include chemical as well as physical changes.

Many processes of preparation are purely physical **changes of form,** such as comminution, sifting, drying, fusion, sublimation, solution, crystallization, turbidation, physical precipitation, granulation, etc.

Other physical processes of preparation are **methods of separation** or extraction, by which one or more of the component ingredients of mixtures are separated. The means employed for this purpose include fusion, sublimation, distillation, solution, filtration, crystallization, physical precipitation, and other methods.

Processes of purification are, of course, also essentially "methods of separation" of different kinds of matter from each other; but distinction may well be made between *a mixture of two or more substances,* and a crude product or natural *material consisting almost wholly of one substance* contaminated with small proportions of other substances which are regarded as "impurities." Processes of purification are frequently purely *physical* processes; but they are much more frequently *chemical* processes.

Processes of production of chemical substances are always chemical processes resulting in the formation of new molecules. But all chemical processes include physical operations.

2. To recognize clearly how the processes employed in the laboratory may or may not be accompanied by chemical reactions the student is invited to note the differences between: 1, the

purely *physical process of sublimation* by which the volatile benzoic acid contained in benzoin is separated from the non-volatile resin and other fixed substances with which it is associated, and the *chemical sublimation* by which mercuric chloride is produced from a mixture of mercuric sulphate and sodium chloride; 2, the purely *physical process of trituration* by which any single substance may be reduced to powder, and the *trituration* by means of which mercury and iodine are brought into contact with each other for the purpose of causing them *to unite chemically* to form mercurous iodide; 3, the purely *physical process of fusion* by which metallic bismuth is liquefied so that it may be run off from accompanying infusible minerals, and the *fusion to induce chemical combination* by which iodine and arsenic are melted together to produce iodide of arsenic; 4, the *simple distillation* by which the ether contained in a mixture of alcohol and ether may be to a great extent separated from the alcohol, and the *chemical distillation* by which ether is *produced* and separated by distilling it from a mixture of alcohol and sulphuric acid; 5, the *simple solution* by which zinc chloride is dissolved in water, and the *chemical solution* by which metallic zinc is dissolved in hydrochloric acid to form zinc chloride; and 6, the *physical precipitation* by which alum is *separated* from its water-solution by the addition of alcohol, and the *chemical precipitation* by which aluminum hydroxide is *produced* when an alum solution is mixed with a solution of sodium carbonate.

3. All the various processes of preparation, separation, purification, or production of chemicals may be conveniently classified into: 1, dry processes; and 2, wet processes.

Dry processes are those in which the materials employed are not liquid at the ordinary room temperatures, nor dissolved in or mixed with any liquid.

The *materials* employed in dry processes are accordingly either solids or gases; *but the solids may be liquefied by fusion or the gases liquefied by condensation.* In most dry processes the materials are exclusively solids.

The *products* obtained by dry processes may be either solids, liquids or gases; but they are in most cases solids separable by fusion or sublimation, or solids and gases.

Dry processes include trituration, fusion, sublimation, dehydration, ignition, calcination, roasting, dry distillation.

Wet processes are those in which the *materials* employed include liquids, whether the liquid or liquids employed constitute chemical factors or are used as simple solvents or other physical media to promote the attainment of the ends sought.

The *products* of wet processes may be solids, liquids, or gases.

The wet processes include solution, crystallization, precipitation, distillation, and various other methods.

4. The **choice of method** must be determined by: 1, the nature of the product sought; 2, the materials available for its production; 3, the nature of the bye-products, if any; and 4, the chemical laws governing the behavior of factors and products in all cases where the results depend upon chemical changes or reactions.

Among the most important facts to be considered are the state of cohesion, relative water-solubility and relative volatility of the products and materials; then the various chemical reactions which may be utilized to convert the different available materials into the products desired, and the separability of the several products from each other when more than one product is formed.

5. Most of the chemical products are **solids.** For the purposes of this treatise we shall classify the solid chemical products into *soluble solids* and *insoluble solids;* into *volatile solids* and *non-volatile solids;* and into *fusible solids* and *infusible solids.*

The **water-soluble metallic salts** are very generally produced from the corresponding acids. Thus the water-soluble metallic nitrates are generally made from nitric acid, sulphates from sulphuric acid, chlorides from hydrochloric acid, acetates from acetic acid, and so on. The processes employed for this purpose are *chemical solution, saturation* or *neutralization,* followed by the requisite method of separation of the products from the bye-products. The materials required in addition to the acids are the metals themselves or their oxides, hydroxides or carbonates or other compounds yielding unobjectionable bye-products—water or gases.

The water-soluble metallic compounds may also be made by *double decomposition* between factors producing insoluble bye-products.

Insoluble metallic compounds are most frequently made by *precipitation,* the materials employed being water-solutions of the required factors and the bye-products being water-soluble.

Volatile solids may be made by *sublimation* if no volatile bye-product be formed.

Non-volatile solids, when not produced by chemical solution or by precipitation, may be made by various dry processes, including double decomposition between dry materials if the bye-product be volatile so that it may be eliminated by sublimation or by dissipation with the aid of heat.

Fusible solids are frequently made by fusion when the bye-product, if any, is infusible so that separation is practicable.

Liquid products are generally produced by chemical distillation, or by chemical solution.

6. Gases are generally produced by metathesis, or by dissociation, at high temperatures.

7. From the facts mentioned in the preceding paragraphs the student will note that the **separability of the products** is an extremely important factor in the selection of methods for the production of chemicals. Assuming that two products are formed (which is generally the case), they may, of course, be readily separated and the process thus rendered practicable: 1, if one product be soluble and the other insoluble; 2, if one product be non-volatile and the other volatile; 3, if one is a fusible solid and the other infusible; 4, if one be a gas and the other a solid or liquid at the temperature of the reaction; and 5, if one product be water or some other liquid which is unobjectionable or easily separated from the other product.

8. But two or more soluble salts contained together in one solution or mixture may frequently be separated from each other (6) if they differ materially in their respective ratios of solubility in the same solvent at any conveniently attainable temperature; or (7) if one be soluble without alteration, and the other insoluble, in another liquid miscible without chemical reaction with the common solvent for both.

Thus if A and B be both contained in nearly equal proportions in solution in the same water and if A be freely soluble while B is only sparingly soluble, it follows that when the solution is concentrated by evaporation B must separate from the solution before A. And if A and B be both contained in solution in the same water and if A be insoluble in alcohol and in diluted alcohol while B is soluble in either, then A must be precipitated on the addition of alcohol while B remains in the liquid.

9. The prognosis of the reactions which will probably take place between the factors brought into contact with each other in any chemical process is generally rendered practicable by a good knowledge of the conditions which are known to affect their direction and relative completeness, such as: 1, the quality, quantity and intensity of the chemical combining power of the elements composing the factors of the reaction, including the greater energy of radicals in the nascent state; 2, the influence of predisposing affinity; 3, the freedom of contact between the factors, including the removal of one of the products from the scene of action and the influence in that direction of the cohesion, solubility, fusibility and volatility of the products; 4, the influence of temperature; and 5, the relative masses of the reacting substances.

These matters were discussed in Chapter XIX of Vol. I.

10. The different forms of chemical reaction were fully described in Chapter XVII of Vol. I. The most common reactions by which inorganic chemical products are formed are: *Dissociation,* or decomposition; *Synthesis,* or combination or composition; *Metathesis,* or double decomposition, including *Substitution;* and reactions of *Oxidation and Reduction,* or reactions involving changes of atomic polarity-value.

11. The choice of materials. The factors or materials that can be used for the production of chemical preparations may generally be any substances containing or furnishing the elements composing the product sought. Thus any compound of mercury can be made out of any other compound of mercury, and any iodide can be employed for the preparation of any other iodine compound.

But the best materials are those that give satisfactory results with the least expenditure of time and labor and at the least cost. Hence we would not make mercuric iodide out of mercuric sulphate, nor out of aristol or any other expensive or complex iodine compound.

The materials necessary are generally in any given case, two: one of them to contribute the positive radical and the other to contribute the negative radical of the product sought.

There are usually several kinds of inexpensive materials available; but the cheapest materials frequently demand the most tedious and expensive processes, while easy and inexpensive methods are generally applicable when the materials are of a

higher grade. Cheap raw materials are, therefore, used only in manufacturing on a large scale with all the labor-saving devices and special facilities requisite to obtain satisfactory results at the least cost.

To prepare pharmaceutical chemicals and other *pure chemical products* it is necessary to employ, as far as practicable, materials of definite composition and free from any impurities that can not be easily removed in the process adopted. Unfit materials furnish unfit products.

12. The most common materials employed for the production of pure inorganic chemicals are:

Acids and their solutions.

Alkalies and their solutions.

The metals.

Metallic oxides.

Metallic hydroxides.

Metallic carbonates.

Soluble metallic sulphates, nitrates, phosphates, acetates and other soluble metallic oxygen salts.

Soluble metallic chlorides, bromides and iodides.

The non-metallic elements chlorine, bromine, iodine, sulphur and carbon.

13. Whenever solutions of acids, alkalies, or salts, or other solutions are employed as materials, it is necessary that their strength shall be exactly determined and that the relative proportions used be governed accordingly, and the quantities prescribed in stated formulas must be corrected in every instance as required.

The *actual strength* of acids, ammonia water, and other solutions frequently varies materially from the official or commonly recognized standards and from the strength specified on the label or indicated by the title.

If the materials be crystallized substances containing water of crystallization they must be in perfect condition, either containing the known full amount of such water, or dried until they cease to lose weight or attain a definite composition in accordance with which the proportion required for the reaction may be exactly determined. It is clear if 100 Gm. of crystallized sodium carbonate is required, that 100 Gm. of effloresced sodium carbonate must be too much; and that if one kilogram of an anhydrous salt

is required the same quantity of a salt containing one molecule of water of crystallization is insufficient, and a kilo of a salt containing several molecules of crystal water must be still farther from the correct amount.

Whenever hygroscopic substances are used they must be perfectly dry; or, if the moisture be unobjectionable, its exact proportion must be known and taken into account, for it one pound of dry potassium carbonate or potassium cyanide is required, one pound of wet carbonate or cyanide will not be enough.

14. *Acids* employed in the laboratory must be tested both qualitatively and quantitatively before they can be safely used. Their strength can not be sufficiently accurately determined by their respective specific weights; it must be ascertained by volumetric analysis. Should it be found to differ from that upon which the proportions in the working formula are based, those proportions must be corrected to correspond with the acid used or the strength of the acid must be changed to accord with the formula.

If the Pharmacopœia prescribes a sulphuric acid of "not less than 92.5 per cent" strength, and bases all its working formulas in which that acid is prescribed upon the assumption that the acid employed contains neither more nor less than 92.5 per cent of absolute H_2SO_4, then the sulphuric acid usually sold may frequently be found of such strength as to require the correction of the proportional amount ordered.

Other chemical solutions must be tested in the same manner—by volumetric assay—and the quantity used made to conform to the actual strength found.

15. The water-soluble salts of any metal are most frequently made out of insoluble compounds of that metal if not from the metal itself, and insoluble metallic compounds are generally produced from soluble compounds.

CHAPTER II.

CRUSHING AND POWDERING.

16. It is frequently necessary to **crush** solid substances preparatory to their use as materials.

A large piece may be broken on the anvil with a hammer, or in an iron mortar by well directed blows with the pestle. To prevent the fragments from being scattered the piece may first be wrapped in several thicknesses of strong paper.

Crushing machines or iron *mills* are useful for coarse comminution when the substance to be crushed or coarsely ground does not consist of or contain pieces too large to be put through the machine.

Hard and tough minerals like manganese dioxide in lumps, hæmatite, iron sulphide, antimony sulphide, marble, and other materials used in considerable quantities, may be advantageously broken, crushed, and even powdered, in a deep iron mortar.

17. Some minerals which are too unyielding to be crushed and powdered in the iron mortar without other aids may be heated to dull redness, or near it, and then, while hot, dropped into cold water. This treatment sometimes renders such substances much more tractable. Hæmatite can be reduced in that manner. This plan is, of course, not applicable to substances which are decomposed or fused by the high temperature.

18. The **iron mortar** must be large and deep, and the pestle heavy enough to do a considerable portion of the work of contusion by its own weight.

Small mortars are practically useless for such purposes as the crushing and pulverization of hard and tough substances, because the quantity operated upon at one time must not be greater than that barely sufficient to cover the bottom of the mortar to the depth of about 10 to 50 millimeters, according to its size.

An iron mortar about 0.50 meter deep and 0.25 meter in diameter is perhaps most useful in the average laboratory, being sufficient for the effective contusion of as large quantities as can be conveniently treated all at once, and not too large for much

smaller quantities. When a very large quantity of any material is to be crushed or powdered it is, of course, necessary to divide it into portions not too large for effective work.

A larger iron mortar is probably too heavy for general laboratory use, but an additional mortar of about one-third the dimensions specified will be found useful occasionally.

Being very heavy the large iron mortar should be placed firmly upon a solid foundation, preferably on a wooden block fixed in the ground and resting on a large stone.

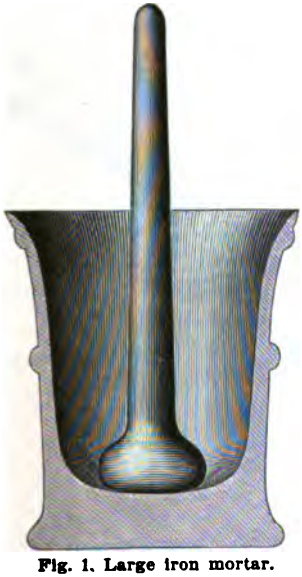


Fig. 1. Large iron mortar.



Fig. 2. Small iron mortar.

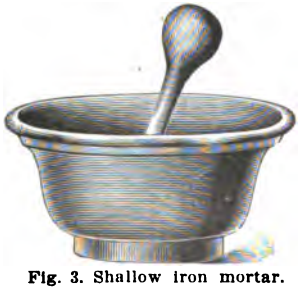


Fig. 3. Shallow iron mortar.

The iron mortar and pestle are more generally useful and effective than any crushing machines or mills for ordinary operations. Insoluble solids rarely attack iron and may be powdered in the iron mortar without becoming contaminated with iron. Even soluble chemicals may frequently be safely permitted to come in contact with iron; but the mortar and pestle must be at all times kept bright and clean—quite free from rust.

Perfectly dry chemicals can usually be safely pulverized in a bright, dry iron mortar; but moist chemicals and such as contain

water of crystallization should be powdered in porcelain mortars.

Very brittle or friable substances that can be easily crushed and reduced to powder by trituration in a porcelain mortar should not be powdered in the iron mortar; nor should any acid or alkali,



Fig. 4. Steel spatula used with small iron mortars and iron dishes.

or other substance which may act chemically upon the iron, be placed in it.

19. Porcelain mortars for crushing and powdering chemicals, and for mixing powdered materials, should, like the iron mortar, be very capacious in proportion to the quantities triturated at



Fig. 5. Trituration mortar of porcelain.

one time in them, but not so deep. Several sizes of porcelain mortars are required, varying from 100 to 400 millimeters in diameter and of a depth equal to about two-thirds of the diameter. It is very important that every mortar shall have a regular, spherically concave, smooth bottom, free from "nipples" and "wrinkles," or from elevations or irregular depressions of any kind, and that its pestle shall fit it perfectly. The convex head of the pestle must be large and perfectly spherical, but of a slightly smaller radius than that of the curved bottom or grinding surface of the mortar. The handle of the pestle must be large and strong so that it can be firmly grasped with the whole hand

in order that sufficient pressure may be conveniently exercised in the act of triturating.

Effective trituration is impracticable when the quantity of matêrial contained in the mortar is too great, and the best results are attained when the layer of powder is not greater than that required to well cover the bottom of the mortar.

When large crystals are to be broken or crushed in the porcelain mortar, sufficient care should be exercised to prevent the scattering of the fragments, and it is sometimes best to wrap the large crystal or piece in muslin or paper.



Fig. 6. Small porcelain mortar.

Only comparatively brittle substances can be easily powdered by trituration.

20. When *dry poisonous substances* are triturated in a mortar, care must be taken to prevent the danger from dust rising out of the mortar. It is sometimes advisable to moisten the contents of the mortar with water or alcohol if admissible. Arsenous oxide and mercuric oxide are good examples of substances that ought not to be triturated or sifted in a dry condition if considerable quantities are thus treated so that the operations are too extensive and extended to be safe.

21. The *spatula* is indispensable in employing trituration as a means of reducing substances to fine powder and in mixing powdered materials, for most substances have a tendency to adhere



Fig. 7. Steel spatula.

more or less to the surfaces of mortars and pestles when triturated under pressure, and must be scraped off with the spatula.

22. *Levigation* is the trituration of a powder to an extremely fine state of division on a slab with a muller, adding a liquid, as either water or oil, to form a very soft paste with the powder. But the levigation may also be very advantageously effected in a perfect mortar with a perfect pestle, instead of using a slab and muller.

23. Sometimes levigation is followed by *elutriation.* This process consists in agitating finely powdered insoluble substances with large quantities of water, then permitting the coarser par-

ticles to subside during a brief cessation of the agitation, after which the liquid, holding the finest powder still in suspension, is decanted into another vessel. By repeated alternate levigation and elutriation the product may thus be reduced to an "impalpable" or extremely fine powder. Prepared chalk and purified antimony sulphide are thus treated.

24. Dry substances which have been powdered by grinding, contusion or trituration require *sifting* if it is necessary to render their fineness uniform or to remove any coarse particles the powders may contain. The *sieves* may be made of hair cloth, silk, or brass wire gauze. The finest powders are sifted through bolting cloth of silk. Insoluble inorganic substances should be reduced to the finest powder attainable, and should in no case be coarser than No. 80.

The numbers employed to designate the fineness of powders are also applied to the sieves, and they refer to the meshes or openings to each linear inch. Thus a No. 80 sieve is one having 80 meshes to each linear inch of the sieve cloth, and a No. 80 powder is one that has been passed through such a sieve. It were better if the diameter of each mesh were given for the meshes are not always square and the wire, hair, or thread, not always of the same calibre.

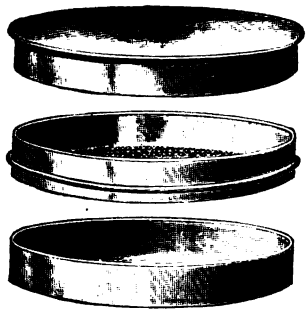


Fig. 8. Metal sieve with covers, or drum sieve.

As finely powdered inorganic substances are generally light enough to rise into the air when sifted in an open sieve, making the operation very troublesome on account of the dust, it is customary to use *drum sieves,* or sieves provided with covers above and receptacles below. These covers are made of drum skin in wooden frames like those in which the sieve cloth is placed, or the sieve frame and covers are made of brass or of tinned iron. For sifting insoluble inorganic substances the drum sieves made wholly of metal are generally most useful.

25. Very friable soft masses of dried precipitates, such as those of magnesium carbonate, bismuth subnitrate, bismuth subcarbonate, etc., can be powdered by *gently* rubbing them through

the sieve cloth with a comparatively soft brush, or by rubbing the pieces against the brass wire cloth in the sieve.

Powders should never be *forced* through the sieve if a soft and fine product is desired, and a fine soft powder should be made in every case where it is possible.

26. Zinc, tin, and some other readily fusible metals may be reduced to small pieces, or "granulated," by pouring the melted metal into a pan or bucket of water.

27. Coarse powder or granular products may be obtained by precipitation, granulation, and turbidation, described elsewhere.

CHAPTER III.

DRY CHEMICAL PROCESSES.

28. Reactions induced by trituration. A few chemical compounds and pharmaceutical chemicals of indefinite composition can be produced by triturating the dry materials together. Long continued trituration and strong pressure are necessary in such cases. Chemical reactions brought about in this way are generally incomplete, and the products accordingly impure and unsatisfactory.

Anhydrous substances and many other perfectly dry chemicals may sometimes be intimately mixed with each other by light trituration without any chemical reaction; this happens, too, in cases where at least partial reaction takes place between the same materials if triturated together under strong pressure or if water is added.

Crystallized hydrous salts when triturated together may react upon each other and a wet mixture may be obtained without the addition of any moisture on account of the liberation of the water of crystallization.

When dry solids unite chemically under strong trituration the synthesis may develop so high a temperature that means must be taken to control it. Thus alcohol is added when mercury and iodine are rubbed together to form iodide of mercury; the evaporation of the alcohol then lowers the temperature and prevents the loss of iodine by vaporization.

29. Fusion is frequently resorted to in chemical operations, sometimes simply to separate fusible from infusible substances, but in other cases to induce chemical reactions between the materials fused together.

Whenever the object is to induce chemical reaction the materials should, if practicable, first be powdered and well mixed. The temperature required to produce fusion is obtained partly from the heat applied to start the reaction and partly from the heat generated by the reaction itself when once started.

The vessels in which substances are fused differ widely, accord-

ing to the temperature necessary. The fusion may in some cases be accomplished in glass beakers or bottles, or in porcelain dishes or crucibles, at temperatures below the boiling point of water; in other cases "red heat," or even "white heat," is required, and crucibles must then be used which are made of materials such as can be subjected to that high heat without risk. The principal materials out of which such crucibles are made include fire-clay, graphite, and platinum.

Clay crucibles are more or less porous and are also liable to crack; accordingly they can not be used more than once except for the same substances, and then only two or three times. Graphite crucibles do not crack so easily, nor are they porous; but they are soon burnt out. A combination of clay and graphite is



Fig. 9 and 10. Clay crucibles.



Fig. 11. Graphite crucibles.

sometimes used and to good advantage. Platinum crucibles are necessary for many operations; but do not resist the chemical action of certain substances.

When fusion is effected it is to be remembered that so long as any of the fusible solid substance remains to be liquefied the temperature of the contents of the vessel in which the fusion is performed remains practically stationary; but that, as soon as all of the fusible matter has been reduced to the liquid state, the temperature may rise very rapidly if the application of strong heat from without be continued, and that in cases where a too high temperature is liable to do damage, it may be necessary to promptly remove the crucible or other vessel from the fire or to discontinue the heating before any damage is done.

Fusion is exemplified in processes of purification of bismuth, antimony sulphide, and many other substances, and in the preparation of moulded and diluted silver nitrate, fused calcium chloride,

granulated zinc, the iodides of arsenic and sulphur, potassium cyanide and sulphurated potassa.

Distinction is to be made between true fusion (sometimes called "igneous fusion," when accomplished at a very high tempera-

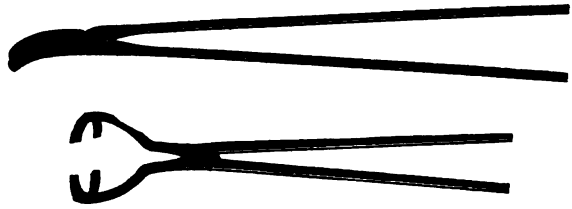


Fig. 12 and 13. Crucible tongs.

ture), and the solution of hydrous crystallized salts in their own water of crystallization when heated (which is called "aqueous fusion)." See par. 33.

30. Ignition without fusion always has for its object a greater or less change in the composition of the substance heated. These changes include the expulsion of volatile impurities, the expulsion of water, dissociation, oxidation, and various intramolecular rearrangements of the atomic linking.

Ignition is effected in iron pots, clay crucibles, and other vessels, and generally at very high temperatures. The substances to be subjected to ignition are, if practicable, first powdered, and when two or more substances are to be ignited together they are first mixed with each other as intimately as possible.

Exsiccation, calcination, and dry oxidation are examples of ignition.

31. Dehydration of solids. The removal of water held by chemical products is called dehydration. It is effected by various methods of drying.

Hygroscopic moisture may be expelled from solids by **desiccation.** The heat applied for this purpose must be sufficient to accomplish the object without unnecessary loss of time, but not so high that the product may be injured or lost by decomposition or volatilization. The solid substance in coarse or fine powder or in a granular or crystalline condition, according to its nature, is spread out over the bottom of a shallow dish and carefully heated. It must be constantly or frequently stirred during the process of desiccation, and when quite dry the product should be at once put

into prefectly dry, warm containers, which must be tightly closed.

Some solids must be kept in fusion for some time in order to render them anhydrous.

Unstable or volatile solids when moist may often be successfully dried over lime, sulphuric acid, or other substances having a great avidity for water. This method of desiccation will be described later.

Water of crystallization is eliminated by *efflorescence* or by *exsiccation.*

32. The partial or complete removal of water of crystallization at common room temperatures, or its gradual expulsion by moderate heat, is called **efflorescence.**

Substances which crystallize with several molecules of water do not hold all of that water with equal force. Thus many salts lose a portion of the water of crystallization at about 20° or 35° to 40° or 60°, but retain the remainder at higher temperatures. (See also par. 34.)

Several substances which give up a portion of their water of crystallization at the common room temperatures *in dry air* do not effloresce at the same temperatures *in moist air.*

When the gradual efflorescence of a salt is desired the temperature must be regulated according to the known results of experience with that particular salt, for no general rule is applicable to different salts.

Large crystals should be broken into small fragments before being exposed to the air to effloresce.

The efflorescence should be effected in a current of dry, warm air, and the substance subjected to this treatment should be spread out in a thin layer and occasionally stirred.

Drying closets are necessary in all laboratories where chemical products are made in considerable quantities.

33. Some readily water-soluble substances in the form of crystals containing much water of crystallization dissolve in their crystal-water when heated They are then said to undergo *aqueous fusion.* Alum, sodium phosphate, sodium carbonate, ferrous sulphate, sodium sulphate, and several other salts may be easily dissolved in their own water of crystallization by heating the crystals. When the temperature is raised above that required to cause aqueous fusion, the salt begins to give up its crystal-water, and

finally becomes dry although it may still retain some molecularly combined water if the temperature is insufficient to expel all.

34. The amount of water of crystallization expelled by heat depends upon the temperature. If the substance contains two or more molecules of water it may give up one or more molecules at one given temperature, and other molecules of it at certain definite higher temperatures.

Sodium arsenate loses all of its seven molecules of water at about 149°; but five molecules of it are expelled by complete efflorescence at a gentle heat.

35. Exsiccation is a term used to signify the expulsion of water of crystallization by the aid of heat.

Dried sodium carbonate, sodium sulphate, sodium phosphate, alum, ferrous sulphate, magnesium sulphate, and several other exsiccated salts are common preparations.

Such preparations are not always anhydrous, but each exsiccated salt if not anhydrous must contain a definite amount of water.

The "dried alum" of the Pharmacopœia must be anhydrous; "dried ferrous sulphate" must consist of two molecules of FeH_2SO_5 with not over one molecule of water; "dried sodium carbonate" of the Pharmacopœia contains two molecules of water; and anhydrous sodium carbonate is also used.

In order to obtain products of definite composition by exsiccation it is necessary that the crystallized or effloresced solids shall be subjected to a fixed degree of heat until they cease to lose weight.

It is generally (but not always) advantageous to reduce crystallized salts to coarse powder before they are subjected to exsiccation. In some cases a softer product is obtained when the crystallized salt is first effloresced at a temperature sufficiently low to prevent aqueous fusion, and the exsiccation completed at the lowest temperature sufficient to expel all of the water which it is desired to eliminate.

In all operations of efflorescence and exsiccation it is important that the product be well protected against contamination by dust.

36. Dry dissociation of chemical compounds yielding gaseous decomposition-products occurs in various laboratory processes. The production of oxygen by heating either potassium chlorate or mercuric oxide, and other dry gas-operations, and the conver-

sion of phosphates into pyrophosphates, illustrate the decomposition of chemical compounds in a solid state by heat.

But the most common example of dry dissociation by heat is the production of metallic oxides by strongly heating the hydroxides, nitrates, oxalates, sulphates or other salts affording volatile bye-products. This is called **calcination,** and it is so named because lime (*calx*) or calcium oxide is obtained by strongly heating calcium carbonate in lime kilns.

Among the metallic oxides that may be prepared by calcination are: *magnesium* oxide, from the hydroxide or the carbonate; the oxides of *calcium, strontium* and *barium,* from the hydroxides or the carbonates; *ferric* oxide, from the hydroxide or the oxalate; *cupric* oxide, from the hydroxide or the nitrate; *zinc* oxide, from the hydroxide or the carbonate; *mercuric* oxide, from the nitrate; *lead* oxide, from the hydroxide or the carbonate or nitrate; and *bismuth* oxide, from the hydroxide.

37. Dry oxidation is often effected by the *combustion* of metals in free access of air, or by the *ignition* of reducing agents with oxidizing agents, or by *roasting* metallic sulphides.

Among the metallic oxides that can be produced by the oxidation of the metals in air we have magnesium oxide, zinc oxide, lead oxide and mercuric oxide.

The oxides of zinc, lead, antimony and some other metals can be made by "roasting" their sulphides in the air.

Arsenites may be oxidized to arsenates by ignition with nitrates, and other analogous dry oxidations may be effected with various oxidizing agents such as manganese dioxide, potassium dichromate, and lead dioxide.

38. Sublimation. Volatile solids may be vaporized and the vapor condensed to the solid state again either in the upper and cooler portions of the same vessels in which the solid substances are heated, or in suitable receivers connected therewith.

The vessels employed are special flasks or retorts of glass, earthenware or metal, and the condensing receptacles, if any are required, may be cones or chambers of the same materials or of paper, etc.

Calomel, mercuric chloride, iodine, mercuric sulphide, arsenous oxide, ammonium chloride, ammonium carbonate, and certain other substances may be prepared or purified by sublimation.

In the separation of volatile solids from fixed solids by means

of heat, the volatile substance may be an impurity which it may or may not be worth while to collect by condensation, or the volatile substance may be the chief product and the fixed substances impurities.

The condensed volatile solid is called a *sublimate.*

In processes of sublimation the condenser is usually closer to the vessel in which the vapor is produced than is the case in distillation, because the difference between the temperature to which the vapor is heated and that at which it is condensed is not great.

The sublimate may be in the form of fine powder, as in the case of calomel, or in large aggregated crystals or crystalline masses, as in corrosive sublimate, or in large amorphous masses, as in arsenous acid when manufactured on a large scale.

When the sublimate must be in a finely divided condition the vessel in which the vapor is formed must be kept at a sufficiently high temperature in every part to prevent any condensation of the vapor before it passes into the condenser, and the condenser must be cooled considerably below that temperature.

But when large masses of sublimate (whether crystalline or amorphous) are desired, the condensation must be effected at a temperature but little lower than that of the vaporization of the solid, and in such cases the product is frequently collected in the upper part of the same vessel in which the vapor is produced, or the whole apparatus, including connecting tube and condenser, must be kept hot enough to insure the desired result.

When a flask or retort is employed for the vaporization it is necessary to guard against "choking" caused by the accumulation of sublimate in the neck whereby this may be nearly or quite closed. The neck must, therefore, in all processes of sublimation, be quite short, and in some cases it must be occasionally freed from sublimate by raking the latter out.

In some cases it is the best plan, when the quantities operated upon are not too large, to use tall enough cylindrical vessels of suitable material, vaporizing the solid at the bottom of the cylinders and letting the vapor be condensed at the upper end which is to be rather loosely closed with chalk stoppers, paper or clean cotton. The cylinders, tubes, or cylindrical wide-mouthed bottles, used for sublimation are generally placed in an inclined position.

[The student may make a few preliminary experiments in sub-

limation by operating upon very small quantities (say, about 0.50 Gm.) of any of the following named substances: camphor, benzoic acid, iodine, calomel, corrosive sublimate, mercuric iodide, mercuric sulphide, ammonium carbonate, ammonium chloride, arsenous oxide. Use a *long* test-tube; place the substance to be sublimed in the bottom of the tube; use a test-tube holder to prevent burning the fingers; heat the lower third of the tube, especially at the bottom, by moving it up and down in the flame of a Bunsen burner or a spirit lamp, and let the sublimate be condensed in the cooler upper end of the tube. If arsenous oxide, or any mercury compound, or other poisonous substance is experimented with, care must be taken not to permit the escape of the poisonous vapor and its inhalation.]

CHAPTER IV.

SOLUTION—ITS NATURE, CAUSES AND EFFECTS.

39. Numerous striking phenomena of **solution** are familiar to all. Sugar, salt, washing soda and many other solids are completely liquefied when put in a sufficient quantity of water, and the result in each case is a perfectly homogeneous liquid.

Pieces of camphor when put in alcohol *dissolve* in that liquid, yielding a uniform solution containing no visible particles of the camphor.

Metallic iron placed in hydrochloric acid disappears as iron and, if a sufficient quantity of acid be used, a liquid is obtained in which no solid particles of matter are visible.

Rosin is taken up in solution by oil of turpentine; glycerin dissolves crystallized carbolic acid; hot olive oil makes a perfect solution with wax; and benzin forms with lard an entirely homogeneous liquid.

These phenomena of the *solution of solids in liquids* are more striking than solutions of liquids in each other, solutions of gases in liquids, and other instances of intimate molecular blending of two or more substances, because we see that the pieces or particles of solids gradually dissolve and finally disappear in the liquid or solvent.

40. We know further that many solids are unaffected by water and by various other liquids.

Marble does not dissolve in water, alcohol, ether, chloroform, benzin, oils, or glycerin; but it does dissolve in hydrochloric acid, in nitric acid, and in vinegar. Glass is insoluble in nearly all liquids except hydrofluoric acid and strong alkali solutions. Diamond is insoluble in all liquids.

41. Zinc chloride dissolves in less than one-third of its weight of water; potassium hydroxide in about one-half of its weight; potassium iodide in three-fourths of its weight; sodium thiosulphate in two-thirds; Rochelle salt in one and one-half times its weight of water; alum in nine times its weight; baking soda requires nearly twelve times its weight of water to dissolve it;

cream of tartar over two hundred times; lead iodide two thousand times; copper sulphide nearly one million; and silver bromide about two million times its weight of water.

Camphor dissolves in about one-third its weight of chloroform; in its own weight of alcohol; and in from fifteen hundred to two thousand times its weight of water.

42. Lixiviation is a term applied to the separation of soluble inorganic solids from insoluble solid matter by the aid of water.

Potassium carbonate is "leeched" out from wood ashes, and acid calcium phosphate from a mixture made of bone-ash and sulphuric acid, by passing water through the mixed matter. This process, it will be seen, is somewhat analogous to percolation or to circulatory displacement, according to the manner in which it is effected. But lixiviation may also be effected by maceration or digestion of the solid matter with water, the solution formed being then separated from the residue either by decantation or by straining or filtration.

43. Turning now to *the solvent action of liquids upon each other* we find that comparatively few liquids can be mixed with each other in all proportions to form permanent, homogeneous, clear solutions, and that some pairs of liquids can not be mixed in any proportions to form such solutions.

Alcohol and water, water and glycerin, alcohol and glycerin, alcohol and ether, ether and chloroform, and volatile and fixed oils, form perfect solutions in all proportions; but water is not soluble in fixed oils, nor any fixed oil in water.

Water dissolves ten per cent of its weight of ether, ether dissolves three per cent of its weight of water, and these two different solutions—the solution of ether in water and the solution of water in ether—are not miscible with each other or soluble in each other.

Phenol dissolves in about fifteen times its weight of water, and water is soluble in about twelve to thirteen times its weight of phenol; both solutions are clear and permanent at the ordinary room temperatures, but they are immiscible with each other. At 80° C., however, phenol and water are miscible with each other in all proportions, forming clear homogeneous solutions.

Valeric acid is soluble in thirty parts of water, and water is soluble in five parts of valeric acid; these solutions do not dissolve each other.

But any two liquids which dissolve each other in certain definite proportions but not in all proportions can be combined into perfect homogeneous solutions by the addition of a third liquid miscible in all proportions with each of the other two.

44. *Solutions of gases in liquids* are also formed. In fact, all gases are soluble to some extent in all liquids, although it often happens that the gas dissolves in the liquid so sparingly as to be regarded as practically insoluble.

One volume of water dissolves about 1.8 volumes of carbon dioxide; 4.3 volumes of hydrogen sulphide; 79.8 volumes of sulphur dioxide; 503 volumes of hydrochloric acid; 1050 volumes of ammonia.

Any given volume of any given liquid dissolves a given number of volumes of any given gas at any given temperature without regard to the pressure. Thus one volume of water dissolves 1.8 volumes of carbon dioxide not only at the ordinary atmospheric pressure, but also under twice that pressure, or three or any other number of times that pressure; but 1.8 volumes of carbon dioxide under the pressure of two atmospheres weighs twice as much or is twice as large a quantity of carbon dioxide as the same number of volumes of that gas under the pressure of one atmosphere.

45. For the purposes of this book we shall adhere to the generally recognized **definition of solution**: the liquefaction of any substance by the action upon it of any liquid, the product being a homogeneous clear liquid made up of all of the liquid used as a solvent and all of the substance dissolved in it.

This definition recognizes no solvent except a liquid solvent, and no solution except a liquid solution.

But in a more general sense we have also solutions of gases in other gases, solutions of gases and solids in each other, and products of the process of solution which are not liquid. The evaporation of a liquid into the air may be looked upon as a solution of the liquid in the gases of which the air consists.

46. A real or *perfect solution* is, accordingly, for the purposes of practical pharmacy and pharmaceutical chemistry, a perfectly homogeneous and clear liquid consisting of one substance originally in liquid form recognized as the solvent, and one or more other substances which may have been, previously to their

solution, either solid, liquid, or gaseous, but which are contained in the solution in a liquid form.

47. Solutions can, in many cases, be separated again into their original constituents. Thus a solution of salt in water can be boiled down to dryness, the water being thus eliminated by vaporization, leaving the whole quantity of the salt in its original solid state and with its characteristic properties unchanged.

Water can also be at least partially separated from solutions by freezing, for only the water enters into the formation of the ice.

A solution made of glycerin and alcohol can be separated into its original components by distilling it, the volatile alcohol forming the distillate while the non-volatile glycerin remains in the still or flask. A solution of ammonia in water can be more or less completely separated into H_3N and H_2O by heating it whereby the ammonia is expelled.

But when iron is dissolved in hydrochloric acid, or zinc in sulphuric acid, the metal can not be recovered from the solution, nor the acid separated from it, by any such simple means, because these solutions do not consist of the original materials put together, but of different substances formed by chemical reaction between the original substances.

48. **Simple solution** is to be distinguished from chemical solution by the fact that in simple solution the original molecules of the solvent and dissolved matter remain unaltered and no new molecules are formed, while

Chemical solution results in the formation of new molecules which take the place of the original substances.

It is self-evident, however, that chemical solution is a chemical reaction in which one of the factors is a liquid and resulting in products which form together a homogeneous liquid by solution. In other words, chemical solution is simple solution preceded by a chemical reaction between factors of which one is a liquid.

Examples of simple solution are very numerous for nearly all solutions in which water, alcohol, a volatile oil, or a hydrocarbon is the solvent, are simple solutions.

Among the most common examples of chemical solution are: the solution of metals, metallic oxides and hydroxides, and other metallic compounds, in acids, the solution of bromine or iodine, or sulphur, in alkali solutions; the solution of iron and bromine, or of iron and iodine, together, in water; the official processes

for the preparation of solution of potassium arsenite, solution of subacetate of lead, etc.

49. Simple solvents, or neutral solvents, are liquids which dissolve soluble substances without chemical change.

Chemical solvents are liquids which dissolve substances by transforming them into new and different substances, the original molecules of both solvent and dissolved matter disappearing.

50. *Solution is caused by* some form of *molecular attraction.* It is generally ascribed to adhesion—the attraction between unlike molecules.

51. Mendeleeff describes solutions as "fluid, unstable, definite chemical compounds in a state of dissociation." He says "the conception of solutions as liquid dissociated definite chemical compounds is based on the following considerations: (1) that there exist certain undoubtedly definite chemical crystalline compounds (such as $H_2SO_4.H_2O$, or $NaCl.10H_2O$, or $CaCl_2.6H_2O$, etc.) which melt on a certain rise of temperature, and then form real solutions [see Par. 52]; (2) that metallic alloys in a molten condition are real solutions, but on cooling they often give entirely distinct and definite crystalline compounds, which are recognized by the properties of alloys; (3) that between the solvent and the substance dissolved there are formed, in a number of cases, many undoubtedly definite compounds, such as compounds with water of crystallization; (4) that the physical properties of solutions, and especially their specific gravities (which are very accurately observable) vary with a change in composition, and in such a manner as the formation of one or several definite but dissociating compounds would require. Thus, for example, on adding water to fuming sulphuric acid its density is observed to decrease until it attains the definite composition H_2SO_4, or SO_3+H_2O [see Par. 55], when the specific gravity increases, although on further diluting with water it again falls."

"The two aspects of solution above mentioned, and the hypotheses which have as yet been applied to the examination of solutions, although they have partially different starting points, yet will doubtless in time lead to a general theory of solutions, because the same common laws govern both physical and chemical phenomena, inasmuch as the properties and movements of molecules, which determine physical properties, are dependent on the

movements and properties of the atoms, which determine chemical mutual actions."

52. The student is advised to hold fast to the distinction between physical and chemical phenomena as indicated in the foregoing statement, and to the difference between a molecule consisting of atoms united to each other by one complete system of atomic linking in harmony with the doctrines of polarity and valence, and a combination of unlike molecules held together by some form of molecular attraction evidently independent of the laws of polarity and valence. Every molecule has its own (and only one) unbroken system of atomic linking whereby all of its atoms are held together as one united whole; but in such a compound as $CaCl_2.6H_2O$ it is impossible to discover any link or links between any atom or atoms of the $CaCl_2$ on the one hand and any atom or atoms of the $6H_2O$ on the other, so that the $CaCl_2$ and $6H_2O$ must be held to each other by a *molecular* force quite different from the *atomic* attraction which makes the molecule of $CaCl_2$ a distinct system of atomic linking and each molecule of H_2O another and equally independent system.

Moreover the student must remember that a salt combined with the water called "water of hydration" and that called "water of crystallization" does not differ essentially in its chemical properties from the anhydrous salt, and that a dilute water-solution of any salt is absolutely identical with any other equally dilute water-solution of the same salt, whether the kind of salt used to make the solution be the anhydrous salt or the salt containing a maximum or a minimum amount of water of crystallization. Water-solutions of sodium carbonate, all of them absolutely identical as to strength, composition, and every property, can be made out of either Na_2CO_3, or $Na_2CO_3.H_2O$, or $Na_2CO_3.5H_2O$, or $Na_2CO_3.7H_2O$, or $Na_2CO_3.10H_2O$, or $Na_2CO_3.15H_2O$.

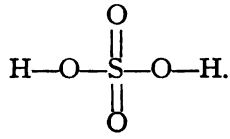
53. All water-solutions contain water as water.

54. Deliquescence indicates a molecular attraction of the deliquescent body for water, resulting in the formation of that kind of a combination or union (of the water and the deliquescent body) which we call a solution.

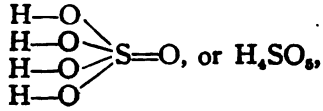
55. But when water is added to SO_3 the result is not a simple solution, but a chemical solution, for H_2SO_4 is formed, which is not



with H—O—H, but



And when H_2SO_4 is brought into contact with another molecule of water the resulting product is not a solution of H_2SO_4 in water, but the chemical compound



which contains no water.

If still another molecule of water be added, the compound H_6SO_6 is formed. This, too, is a true chemical compound, or complete system of atomic linking, and not a solution since it contains only one kind of molecules.

56. We know that some substances are soluble and others insoluble in the same solvent; that different soluble substances differ widely in their respective ratios of solubility in the same solvent; and that a substance may be freely soluble in one solvent, moderately soluble in another, sparingly soluble in a third, and quite insoluble in a fourth solvent. We also know that any given solvent dissolves the same quantity of any given substance under the same conditions. But the law which must specially govern solution and solubilities is not yet understood.

57. That molecular attraction of some kind has to do with solution may be seen from the phenomenon of deliquescence; from the fact that a contraction of volume takes place when miscible liquids are united into solutions; from the upward diffusion of a heavy salt in water-solution through superincumbent pure water; from the fact that when a water-solution of a gas is heated it sometimes happens that water alone is first evaporated until the solution acquires a certain degree of concentration, after which this solution, containing a definite proportion of the

gaseous substance, evaporates without further separation; and from other facts.

The well known fact that HCl can not be evaporated off from its water solution proves that the HCl and H_2O are held together strongly; but it does not prove that they are chemically combined (by any atomic linking).

58. Ostwald supports the conclusion that salts in water-solution are separated into simpler constituents. He calls attention to the fact that aqueous solutions of salts and aqueous solutions of the stronger acids and bases, all of which he calls *salt-solutions* for the sake of brevity, "behave as if they contained a greater number of molecules than corresponds with the formulæ of the dissolved salts." From these considerations, and from the facts of electrolysis, he concludes that salt-solutions contain parts of the molecules, or the **ions**, of the salts. This is in agreement with Mendeleeff's definition of solutions (51). [See also Chapter VI., Par. 131, Vol. I.]

59. The velocity of reactions occurring in solutions may be at once understood upon the theory that the reagents are in a state of dissociation into their respective ions or radicals.

60. The divisibility of matter is most strikingly illustrated by the color reactions which may be produced in certain extremely dilute solutions, and Ostwald points out that *elemental radicals* or ions present in a solution behave quite differently from the *compound radicals* or ions containing the same elements. Thus the negative ion of a chloride, Cl, behaves differently from the negative ion ClO_4 of the perchlorates; the negative ion, S, of the sulphides produces reactions quite different from those produced by SO_4; and the positive ion Co of cobalt salts behaves differently from the negative ion $Co(CN)_6$.

61. "The colors of salt-solutions are essentially the colors of the ions contained therein" (Ostwald).

Very dilute solutions of cupric chloride (containing little of the undissociated compound but many free copper ions) are blue; but concentrated solutions of the same compound are green (because they contain fewer copper ions and much undissociated cupric chloride). This and other examples are mentioned to show that the color of a solution is sometimes due to free ions and sometimes to the undecomposed compound. A solution of chromic anhydride does not contain H_2CrO_4, for, if it did, the

ions of CrO_4 would make it yellow; but it does contain $H_2Cr_2O_7$ and is, therefore, colored red by the ion Cr_2O_7.

An interesting example not mentioned in Ostwald's book is that presented by ferrous and ferric salts. If ferrous chloride and ferric chloride are dissociated into their respective ions by solution it must be admitted that the ions of both chlorides are Fe and Cl; but the green solution of ferrous chloride contains diad ferrous ions while the reddish-brown solution of ferric chloride contains triad ferric ions. It is true that the difference between ferrous iron and ferric iron is known only from their compounds, and that ferrous iron can be oxidized to ferric and ferric iron reduced to ferrous, so that the distinction between diad iron and triad iron must depend upon their respective units of combining value actually used (their respective polarity-values); but the dissociated ferrous and ferric ions in solutions must still be respectively bivalent and trivalent since the removal of any ion from the solution can not be admitted.

The color may depend on either positive or negative ions. If it be admitted that iron salts are in a state of partial dissociation when in water-solution, and that "the colors of salt solutions are essentially the colors of the ions contained therein," then the solutions of ferrous salts are colored green by the ions of diad or ferrous iron, while the solutions of ferric salts are colored reddish-brown by the ions of triad or ferric iron. Yet, ferrous iron and ferric iron are both commonly regarded as single atoms.

[Ferrous and ferric salts in the dry state are generally nearly white or very pale, but they exhibit a decided color as soon as dissolved in water. Other anhydrous salts also exhibit different colors when dissolved in water.]

62. Only acids, bases and salts are believed to undergo dissociation in their water-solutions. If this be true there are numerous substances which form solutions without dissociation. Moreover, the compounds which become separated into their ions by solution in water may dissolve in other liquids without dissociation, strong solutions may contain no free ions, while weaker solutions of the same salt give evidence of partial dissociation; and complete dissociation is not believed to be effected except in infinitely dilute solutions.

CHAPTER V.

SOLVENTS—SOLUBILITY—SOLUTIONS.

63. Common solvents. The most common simple solvents for inorganic substances include water, alcohol, glycerin and acetic acid.

Water is the most important of all solvents, and its value is mainly due to the fact that it is chemically indifferent with relation to most other substances. Accepting the conclusion recently advanced that water, when used as a solvent, causes the partial dissociation of certain classes of water-soluble compounds into their respective ions, we must at the same time remember that the water itself remains undissociated. Water thus facilitates chemical reactions when solutions containing the reagents are mixed, and it is probable that medicinal substances in water-solution must act more promptly and energetically whenever they are in a state of partial dissociation.

Water dissolves a large number of inorganic compounds and also numerous organic substances. But many substances which may long remain unaltered when in solution in alcohol, ether, glycerin, and certain other solvents, are liable to more or less rapid decomposition when kept in water-solution.

Alcohol is an exceedingly valuable pharmaceutical solvent, because, like water, it is generally chemically neutral, it dissolves and extracts from plant drugs most of the constituents that possess medicinal activity, and the solutions it forms are far more permanent than water-solutions. Alcohol, however, dissolves comparatively few inorganic compounds.

Glycerin dissolves perhaps all water-soluble substances and many other substances besides. Glycerin dissolves normal bismuth nitrate without decomposing it. *Glacial acetic acid* also dissolves normal bismuth nitrate.

64. The term **solubility** when mentioned without naming the solvent means the maximum solubility in water at ordinary room temperatures.

The ratio or extent of the solubility of any one substance in

different liquids, and of different substances in the same liquid, may range all the way from absolute insolubility to unlimited solubility. Flint is absolutely insoluble in water and all other simple solvents; but a solution of ferric citrate may be evaporated to complete dryness without any separation of the salt from it.

65. The relation of the solubility of a substance to its chemical composition is not understood. Hence the chemical composition does not furnish a reliable or consistent guide to solubility, for there are many apparent exceptions or inconsistencies.

Thus, while the halides of the three common alkali metals are all readily water-soluble and those of lithium more soluble than those of sodium and potassium, their hydroxides and carbonates stand in the opposite order. Of the halides of calcium, strontium and barium, those of calcium are most freely soluble and those of barium least so, which would seem to agree with the order in which the halides of the alkali metals stand to each other, the metal having the smallest atomic weight forming the most readily soluble halide. But, on the other hand, lead iodide is far more readily soluble than mercuric iodide, while mercuric chloride is far more soluble than lead chloride. The nitrates of copper, silver and lead are all very readily soluble; but while copper sulphate dissolves in 2.6 parts of water, silver sulphate requires 200 parts of water for its solution and lead sulphate 32,000 parts.

Sodium dichromate is more soluble than sodium chromate; but potassium dichromate is less soluble than potassium chromate.

Borax is soluble in 16 parts of water, and cream of tartar in 201 parts; but the two substances together dissolve in a very small amount of water to form "borax-tartar" or potassium-sodium boro-tartrate which is soluble in water without any limit, being deliquescent.

66. The ratio of solubility of a substance may be materially affected by its condition. A hydrous substance is frequently more soluble than the anhydrous; the amorphous substance may be more or less readily soluble than the crystallized; a substance containing more water of crystallization is usually more readily soluble than the same substance with a less percentage of water; a recently prepared chemical compound may be more or less readily soluble than the same substance after it has been kept a long time; dry heat (as, for instance, fusion) may also affect the

solubility of a solid; and solubility may further be affected by contact with other substances.

Crystallized $Al(OH)_3$ is difficultly soluble in acids, while a freshly precipitated amorphous $Al(OH)_3$ is easily soluble in acids but gradually loses its solubility when kept.

67. The presence of one substance in a solution may very greatly affect the solubility of another substance in the same liquid. Thus water-soluble potassium salts are frequently insoluble in saturated solutions of ammonium salts; potassium sulphate is insoluble in a saturated solution of ammonium sulphate, potassium nitrate in a saturated solution of ammonium nitrate, and potassium carbonate in strong ammonia water. Potassium sulphate is insoluble in a saturated solution of potassium carbonate, and potassium hydroxide in a solution of calcium hydroxide.

The total displacement of one salt from its solution by the addition of another salt can sometimes be effected.

68. Organic substances which are soluble in water are frequently found to be nearly or quite insoluble in strong water-solutions of inorganic substances. Thus water-soluble salts of the alkaloids are generally insoluble in concentrated water-solutions of metallic salts; pepsin, soap, and various other substances can be precipitated from their solutions by metallic salts.

69. On the other hand it happens sometimes that the presence of one substance in a solution aids the solution of another substance in the same liquid, as when ammonium chloride facilitates the solution of mercuric chloride.

70. Mixed solvents. When two or more solids are dissolved in a mixture of two or more liquids a separation into distinct layers is sometimes the result.

Although alcohol and water are miscible in all proportions they are sometimes separated from each other on the addition of inorganic salts soluble in water and organic substances soluble in alcohol, so that an aqueous solution of the inorganic salt and an alcoholic solution of the organic compound form separate layers.

But certain single inorganic salts may also cause the partial separation of mixed solvents by forming a separate solution with each. Hydroalcoholic solutions of manganese sulphate, and also of potassium carbonate, may separate into two layers, one of which is a concentrated water-solution containing but little

alcohol, while the other layer consists chiefly of alcohol containing very little of the salt.

71. Immiscible solvents. Water and ether, water and chloroform, and other pairs of liquids which are not soluble in each other in all proportions, are frequently employed for the purpose of transferring certain substances from one solvent to the other, for removing a dissolved substance from its solution, and for separating different substances from each other. A substance dissolved in one of the solvents may be divided between it and the second solvent, or it may be almost completely washed out or withdrawn from its original solution.

72. The partial removal of the solvent from a solution by the addition of another liquid which takes up the solvent with more or less avidity is frequently practicable. Thus water may be partially withdrawn from a strong salt-solution by the careful addition of a superincumbent layer of strong alcohol, when it may happen that the alcohol gradually becomes diluted and the salt is partially separated by crystallization or precipitation from its solution for want of sufficient solvent.

73. By the term **physical precipitation** we mean the separation of a dissolved substance from its solution by the addition of a non-solvent liquid miscible with the solvent.

Since alcohol and water are miscible with each other and many substances which are soluble in one of these liquids are insoluble in the other, various substances in water-solution can be "thrown out of solution" by the addition of alcohol, and many other substances can be, in a similar manner, precipitated from their alcoholic solutions by the addition of water.

A saturated solution of KCl made with pure water at 15° contains 24.6 per cent of the chloride; but if made with water mixed with 10 per cent of alcohol the saturated solution contains only about 20 per cent of the chloride, and if made with a mixture of equal proportions of water and alcohol it contains only about 5 per cent of the salt.

A saturated solution of ferrous sulphate ($FeH_2SO_5.6H_2O$) made with pure water contains 37.2 per cent of the salt (at 15°); but a saturated solution of the same salt made with a mixture of 60 per cent of water and 40 per cent of alcohol contains only 0.3 per cent of the ferrous sulphate.

A saturated water-solution of copper sulphate at 15° contains

27.2 per cent of the salt; but a saturated solution made with a mixture of 60 per cent of water and 40 per cent of alcohol contains only 0.25 per cent of copper sulphate.

74. To aid solution we may employ such means as will bring the solvent into intimate contact with the substance to be dissolved and also any other agencies by which the liquefaction of that substance is facilitated.

To facilitate the solution of a solid the solid substance may be crushed or powdered in order to expose a greater surface to the action of the solvent, and the solid and solvent may be shaken or stirred together to further increase the freedom of



Fig. 14. Solution mortar of porcelain.



Fig. 15. Perforated porcelain basket for dissolving metals in acids by circulatory displacement.

contact between them. If the solid should rest on the bottom of the vessel in which the solution is effected, and the solvent remain at rest over the solid, then the solution first formed must cover and surround the solid so as to prevent fresh portions of solvent from coming in contact with it; but agitation serves the purpose of distributing the solution throughout the whole body of the liquid.

The solid may be placed in a mortar and triturated with successive portions of the solvent, each strong solution thus formed being poured off from the undissolved remainder, and this continued until all of the solid shall have been dissolved. By this means the quantity of solvent required to complete the solution may be reduced, since it is practicable to produce a practically saturated solution with each portion of solvent.

Another effective method is that called *circulatory displacement*, which consists in placing the solid substance in a perforated funnel, dish, or basket, or in a bag, or on a colander or perforated diaphragm, just below the surface of the solvent, so that the solution formed may at once descend to the bottom of the vessel, permitting fresh portions of solvent to freely attack the undissolved remainder of the solid until all of it has entered into solution.

Heat almost always facilitates the solution of solids in liquids because the heat opposes the cohesion which holds the molecules of the solid together.

Solids which have a tendency to form clots or agglutinations when wetted should not be powdered to prepare them for solution. "Scale salts" of iron dissolve much more readily when not crushed or powdered; resins, when dissolved in alcohol or in volatile oils, should be in pieces or in very coarse powder.

Agitation, cold and pressure *facilitate the solution of gases in liquids.*

75. A **contraction of volume** nearly always results from solution, whether the substance dissolved be a solid, a liquid or a gas. In other words, the volume of the resultant solution is nearly always less than the sum of the original volumes of the solvent and the dissolved matter. But the volume of the solution is always greater than the volume of the solvent alone.

When 49.836 volumes of water and 53.939 volumes of absolute alcohol are mixed the total 103.775 volumes contract to form just 100 volumes (at 15° C.).

76. When no chemical action accompanies the process of solution there is always a liberation of heat attendant upon a contraction of volume, and an absorption of heat attendant upon an expansion of volume resulting from the solution.

77. When a salt containing a large amount of water of crystallization is dissolved in water the volume of the solution may exceed the sum of the original volumes of the salt and the water; but when an anhydrous salt capable of taking up much water of crystallization is dissolved in water a contraction of volume may be expected. Contraction of volume occasionally takes place when a concentrated water-solution of a salt is diluted with more water.

78. Relations of heat to solution. Reference has already been made to the liberation of heat due to a contraction of volume in the formation of solutions. But the student should know that

the rise or fall of temperature attendant upon the formation of solutions must be affected also by various other influences. As solids must take up latent heat when they become liquefied by solution, it frequently happens that a very considerable reduction of temperature results when a readily soluble salt is dissolved in water; and as any gas must give up latent heat when liquefied by solution the temperature of the water in which the gas is being dissolved frequently rises perceptibly. Besides, there may be various molecular combinations taking place in the formation of solutions, and these may affect the temperature. Finally, chemical reactions also cause changes of the temperature.

When 17 Gm. of H_3N is compressed to the liquid state it evolves 4400 units of heat; but when it dissolves in 18 Gm. of water the same quantity of H_3N evolves 7535 heat units. The 3135 additional heat units were generated by the chemical reaction by which the H_3N and the H_2O formed H_4NOH.

When 30 parts of absolute alcohol is mixed with 70 parts of water the temperature rises 9.14 degrees, which is not accounted for by the contraction of volume. It is assumed that the alcohol combines in some way with a certain number of molecules of water.

When ether and chloroform are mixed in equal volumes the temperature rises 14.°4.

But when equal volumes of alcohol and carbon disulphide are mixed the temperature falls 5.°6.

That anhydrous compounds which take up water in molecular combination dissolve in water with evolution of heat is well known; but there are many examples of the solution of solids attended by a considerable elevation of temperature in which it is not known what compounds are formed, if any. Great evolution of heat attends the solution of KOH in water; but potassium hydroxide is not known to combine with water to form any definite compound. [No system of atomic linking of K, O and H is possible except KOH.]

Anhydrous calcium chloride takes up water to form a definite hydrate and this hydration is attended by an elevation of temperature; but when hydrous calcium chloride is dissolved in water the temperature falls.

79. Freezing mixtures. When readily water-soluble salts are put in a comparatively small amount of water—say, about twice

their weight—the temperature of the water is greatly depressed by the solution of the salt. The temperature may be reduced as much as 20° by various salts.

A mixture of 3 parts of crystallized calcium chloride and 2 parts of snow will cause mercury to congeal.

Two parts of snow or crushed ice and one part of common salt will make a very effective freezing mixture for ice-cream freezers and for other purposes.

A mixture of 5 parts of ammonium chloride and 5 parts of potassium nitrate with 19 parts of water causes a fall of 20° in the temperature of the water.

80. Heat generally increases the solubility of solids and liquids. Boiling water frequently dissolves many times as much of a metallic salt as can be dissolved in cold water; but any substance which is quite insoluble in cold water is also quite insoluble in hot water.

One hundred parts of boiling water will hold 50 parts of potassium chlorate in solution; but when such a solution is allowed to cool to about 15° only 6 parts of the salt remain in solution while the remaining 44 parts will crystallize out.

Alum is soluble in less than one-third its weight of boiling water, but requires nine times its weight of water at 15° for its solution.

Borax dissolves in one-half its own weight of boiling water, but is not soluble in less than 16 parts of water at 15°.

Eight parts of water at 15° will dissolve no more sodium carbonate than is soluble in 1 part of boiling water.

81. The increased ratio of solubility of a solid in water caused by a rise of the temperature grows more rapidly than the heat rises; but the increased solubility resulting from a continued elevation of temperature is often very irregular.

82. Some solids do not dissolve more readily in hot water than in cold water. This is true of acacia and some other soluble varieties of gum.

Sodium chloride is only slightly more soluble in boiling water than in water at 15°; 100 parts of water at 0° dissolves 35.7 parts of NaCl, 36 parts at 20°, and 39.7 parts at 100°.

A solution of calcium hydroxide made saturated at 15° loses about one-half of the dissolved hydroxide on being boiled (without contact with the air).

83. As the solubility of solids is materially affected by the proportion of water contained in them as water of crystallization or water of hydration, and as this proportion of water may depend upon the temperature, it follows that unexpected differences are sometimes attributable to these facts. It is stated that solutions of sodium sulphate below 33° contain $Na_2SO_4.10H_2O$; but solutions of the same salt having a temperature above 34° contain Na_2SO_4. Hence sodium sulphate dissolves in about one-fourth of its own weight of water at 33°; but its solubility decreases above that temperature so that it requires nearly one-half its own weight of water at 100° to dissolve it.

84. Supersaturated solutions are solutions which at any given temperature retain a larger proportion of the dissolved substance than the solvent is capable of dissolving at that temperature.

We have seen that many salts are more soluble in water at a higher temperature than at a lower temperature. Now, if a saturated solution of such a salt be prepared at any given temperature and then allowed to cool, it frequently happens, if the liquid remains at perfect rest, that all the salt remains in the solution, at least for a time, notwithstanding the reduction of temperature.

If a saturated solution of sodium sulphate be made at 34° it will contain about 80 per cent of the salt and 20 per cent of water; if made at 15° the saturated solution of sodium sulphate will consist of about 26 per cent of the salt and 74 per cent of water. Yet, a solution of sodium sulphate saturated at 34° and then allowed to cool gradually to 15°, if not shaken or otherwise disturbed, will continue to retain in solution 80 per cent of the salt. It is then a supersaturated solution.

A supersaturated solution of the salt at a low temperature may, however, be but a saturated solution of the same salt with a different proportion of water of crystallization.

Hydrous salts generally form supersaturated solutions more readily than anhydrous salts.

Supersaturated solutions are readily formed by sodium sulphate, magnesium sulphate, the alums, and sodium thiosulphate.

85. Determination of the solubility of solids. The solubility of solids in water or in alcohol may be determined by two principal methods: 1, a saturated solution may be prepared at any given temperature and the strength of this solution ascertained; or 2, a saturated solution may be prepared at a higher tempera-

ture, the solution then cooled to any given lower temperature at which it should be kept for a day or two, being occasionally shaken, and its strength then found. The results obtained by the first method are liable to be somewhat too low, and those obtained by the second method are liable to be too high.

86. Solubilities are usually stated at 15°, and this is a very suitable standard temperature for the expression of the solubility of solids, because saturated solutions prepared at 15° are rarely exposed to any lower temperature, the average temperature of the work room being higher.

87. The most satisfactory method of determining the solubility of a salt in water at 15° is to make a saturated solution at about 16° to 20°; to set this solution aside for 24 hours at 15°, shaking it occasionally; then filter; then evaporate a weighed quantity to dryness and weigh the residue.

It is, of course, necessary in carrying out this method to know precisely what the residue is — whether hydrous or anhydrous, and if hydrous, how much water it contains, etc. The result should be verified by analysis; a weighed quantity of the salt being dissolved in a weighed quantity of water and this solution analyzed quantitatively, the result to serve as a check upon the composition of the salt, and also upon the result of a quantitative analysis of the saturated solution, which should also be performed. In other words, the salt used to make the solution, the saturated solution prepared from it, and the residue obtained upon evaporating a given weight of that saturated solution, must all be titrated, and the results compared with the weight of the residue. If the salt is hydrous the residue must be dried at a given temperature until it ceases to lose weight, and, if practicable, *all* of the water of crystallization or any other water contained in it should be expelled.



Fig. 16. Flask for weighing solutions in determinations of solubilities.

88. Dr. Rice's Lysimeter. In order to determine the rate of solubility of a substance at a temperature considerably higher than that of the air in the work room, and to obtain a filtered solution of the same temperature as that at which the solution was saturated, Dr. Charles Rice devised an instrument which he gave

the name "lysimeter," which is here described and shown in fig. 17.

The lysimeter consists of a glass tube, *a*, which is 150 millimeters long and 10 millimeters in external diameter, provided at one end with a well-fitting glass-stopper, *c*, the opposite end of the tube being cup-shaped, with a contraction between the cup and the body of the tube as shown in the cut. A ground glass bell, *e*, is made to fit into the cup. The bottom of this bell is perforated as shown in *f*. The ground glass stopper, *b*, also fits into the cup, and is to be used to close the cup after the removal of the bell.

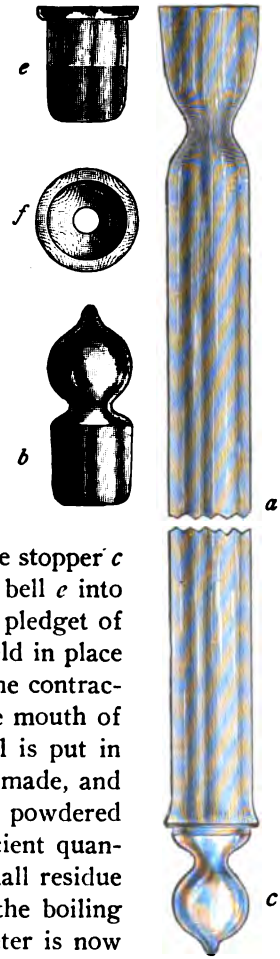


Fig. 17
Dr. Rice's lysimeter.

The quantity of solution must be sufficient so that at least one-half of the instrument can be immersed in it. The solution may be made and contained in a glass cylinder, a beaker, or a wide test-tube. The use of the lysimeter may be illustrated by an example as follows, assuming that the solubility of some solid in boiling alcohol is to be determined: The stopper *c* is inserted into the tube *a*, and the glass bell *e* into the cup-shaped end of the tube. A little pledget of pure cotton is put into the bell, *e*, and held in place by a fine platinum wire passing around the contraction or neck behind the cup and over the mouth of the bell. A sufficient quantity of alcohol is put in the vessel in which the solution is to be made, and heated by immersion in hot water, the powdered solid being added to the alcohol in sufficient quantity to produce a saturated solution, a small residue remaining undissolved after continuing the boiling of the alcohol long enough. The lysimeter is now inserted, prepared as described, cup-shaped end downwards, and when the instrument has assumed the temperature of the boiling liquid, the stopper *c* is removed. The alcoholic solution then enters the tube through the cotton filter in the cup. To insure uniformity of the solution the liquid

may be allowed to flow back through the cotton once or twice. The stopper *c* is now replaced, the instrument withdrawn from the vessel containing the remainder of the solution, and inverted, after which the bell *e* is removed and the stopper *b* inserted in its place. The lysimeter, now closed at both ends by the stoppers, is next washed exteriorly with a little alcohol, and laid aside to cool. The previously ascertained tare of the stoppered tube deducted from the total weight of tube and contents leaves the net weight of the solution contained in it. This is then transferred to a tared dish or beaker, the tube is carefully rinsed with alcohol, the washings being added to the solution, which is then evaporated to dryness over a water-bath, and finally the residue heated to dryness in a drying oven and weighed.

In using the lysimeter with hot solvents the instrument should be gradually heated to the temperature required, as a too sudden change might cause its fracture.

89. Co-efficients of solubility. The number of weight units of any substance required to saturate 100 weight units of water at any given temperature is the co-efficient of solubility of that substance at that temperature.

We have stated that it is the almost universal rule to give the solubilities of solids in water (and in alcohol) at the temperature of 15°, and the numbers expressing their co-efficients of solubility, therefore, refer to and are correct at that temperature only, unless otherwise expressly stated. Thus, when we say that the co-efficient of solubility of borax is 6.25 this statement means that 100 weight of units of water at 15° will dissolve 6.25 weight units of borax to form a saturated solution. The co-efficient of solubility of sodium arsenate is 25 because 25 weight units of the salt $Na_2HAsO_4.7H_2O$ will form a saturated solution with 100 weight units of water at 15°.

This method of expressing ratios of solubility has the advantage that the solubilities of different substances in a constant quantity of solvent are directly represented by the co-efficients so that comparisons are rendered easy and direct. Thus, a substance whose co-efficient of solubility is 10 is twice as soluble as one whose co-efficient is 5, in the same quantity of solvent.

90. Another and more common method of stating the solubilities of substances—a method employed in all pharmacopœias—is that of giving the number of weight units of solvent required to dissolve one weight unit of the substance. Thus the Pharmaco-

pœia tells us that sodium arsenate is soluble in 4 parts of water at 15°, and that borax is soluble in 16 parts. The solubilities of these two substances are *in inverse ratio* as the numbers expressing the respective quantities of solvent required to dissolve them.

91. Both methods of expression are useful, and in laboratory operations we find one of them more direct and convenient in one case, and the other preferable in another case.

In this book we shall make use of both methods according to circumstances, but the student will observe that the co-efficients of solubility do directly express the proportions in which substances can be dissolved, whereas the pharmacopœial method does not directly express the *solubility* of the substance in a given constant quantity of solvent, but the proportion of solvent required to dissolve a constant quantity of the dissolved substance.

92. The student will see that—

The number of weight units of solvent required to dissolve 1 weight unit of the soluble substance is the reciprocal of the number of weight units of the soluble substance required to saturate 1 weight unit of the solvent, and vice versa.

And as

The co-efficient of solubility of any soluble substance is the number of weight units thereof required to saturate 100 weight units of the solvent,

Therefore,

The number of weight units of solvent required to dissolve 1 weight unit of the soluble substance is found by dividing 100 by its co-efficient of solubility, or, in other words, *by multiplying the reciprocal of its co-efficient of solubility by* 100.

And—

The number of weight units of any soluble substance required to saturate 1 weight unit of the solvent is found by dividing the co-efficient of solubility by 100.

93. Methods of fixing and expressing **the strength of solutions.** The strength or degree of concentration of a solution may be fixed or expressed in various ways. Probably no one method is equally convenient for all purposes; but the most generally useful and accurate method is to express the strength of the solution in per cent by weight.

Among the various methods in use are the following:

1. The employment of arbitrary scales of degrees of density

indicated by areometers or hydrometers, such as the scales of Baumé, Twaddell, and others. The strength of salt solutions, syrups, acids, gas solutions, etc., is frequently indicated by some special hydrometer scale.

2. The actual specific weight of a solution may be employed to indicate its relative strength.

3. The strength of a solution may be fixed, as is often done in pharmaceutical preparations, according to the number of customary weight units of the soluble substance contained in any convenient volume of the solution, as, for instance, the number of grains or ounces to each fluidounce, pint or gallon, or the number of grams in each liter.

4. The strength of mixtures of alcohol and water is sometimes expressed in per cent by weight, sometimes in per cent by volume, and sometimes in arbitrary degrees, as in "degrees proof," etc.

5. The strength of gas solutions may be expressed in per cent by weight, or according to the number of volumes of the gas dissolved by each volume of the solvent, and in other ways. It is the custom to state the strength of solution of hydrogen dioxide according to the number of volumes of "available oxygen" obtainable from each volume of the solution.

6. Volumetric solutions are so prepared that each *liter* of finished solution contains as many grams as the number of units expressing the molecular weight of the reagent, divided by the number expressing the valence of either of its two component radicals.

7. Solutions may also be made for laboratory use which contain in each *kilogram* as many grams as the number of units expressing the molecular weight of the dissolved substance, or a convenient decimal proportion thereof.

8. Finally, the strength of solutions may be fixed and expressed in per cent by weight, as is the custom in stating the strength of acids and other solutions of chemicals. This method is the most convenient and scientific, especially if at the same time it includes the principle referred to in the preceding paragraph, so far as applicable or useful.

The molecular weight of HCl being 36.4, it would be more convenient for laboratory purposes to have a solution (hydrochloric acid) containing 36.4 per cent of HCl than to have one containing 31.9 per cent.

CHAPTER VI.

THE CLARIFICATION OF LIQUIDS. STRAINERS. PRESSES. FILTRATION.

94. Unclear liquids are mixtures of liquids with undissolved solid matter, or mixtures of two or more liquids insoluble in each other.

Liquids containing suspended insoluble solid or liquid particles, or mixed with any insoluble matter, generally require clarification before they can be advantageously employed.

The separation of the undissolved substances is effected by some one of the various methods described in this chapter.

95. *Pieces* of solids floating or submerged in otherwise clear liquids may be removed by means of perforated spoons or ladles, or with an ordinary spoon, or by means of pincers. If the solid is heavier than the liquid the latter may be decanted from the solid, or it may be removed by means of a syphon.

Perforated porcelain funnels, such as Buchner's funnel (fig. 18) are very useful for the separation of solids not too minutely divided. Perforated discs which can be placed in ordinary funnels are also much used (fig. 19).



Fig. 18. Buchner funnel of porcelain.

The liquid may also be passed through a piece of coarse, thoroughly washed sponge, or a loose plug of pure ("absorbent") cotton, placed in an ordinary funnel or in a cropped funnel (fig. 20).

96. Clarification by subsidence. Solid matter suspended in a liquid may be allowed to subside, after which the clear liquid is decanted or drawn off. For this purpose the turbid liquid should be put in a tall or deep vessel and left at perfect rest until all of the solid matter has settled to the bottom in as compact a layer of *sediment* as its character may permit it to form. If the solid matter is not too finely divided and if its density is greater than that of the liquid this method of clarification is readily carried out. But when the solid particles are

extremely minute, and especially when their density is about the same as that of the liquid, the operation is slow and difficult if not impossible. It is, however, frequently practicable to sufficiently lower the density of the liquid by dilution or by raising its temperature.

Whenever admissible the clarification may be effected by adding a rather heavy insoluble substance, such as purified clay in powder, which carries the other solid matter down with it as it descends to the bottom of the vessel. The clay or other sufficiently heavy absorbent powder employed must be added in moderate quantity and must be thoroughly distributed through the whole body of the turbid liquid by stirring.



Fig. 19. Perforated disc and its use in an ordinary funnel.

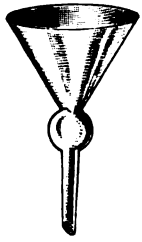


Fig. 20. Cropped funnel.

97. Decantation. A clear liquid standing over a solid deposit of crystals, precipitate or sediment, may be poured off free from solid matter. A complete separation of the liquid by this method is rarely possible, but it may frequently be carried far enough to prove a valuable labor-saving process.

To pour a liquid from one vessel into another without spilling is sometimes a difficult feat, but it is one which every laborant must practice until mastered.

The practical precautions to be observed in this connection are: 1, never to quite fill any vessel with liquid, but to leave enough room to admit of tilting the vessel considerably before any of the

liquid can run over the edge or lip; 2, to use vessels of suitable shape, with a flaring top or rim or provided with a lip; and 3, to handle the vessel with care, deliberately (yet deftly) and with a



Fig. 21. Decantation over a greased rim.

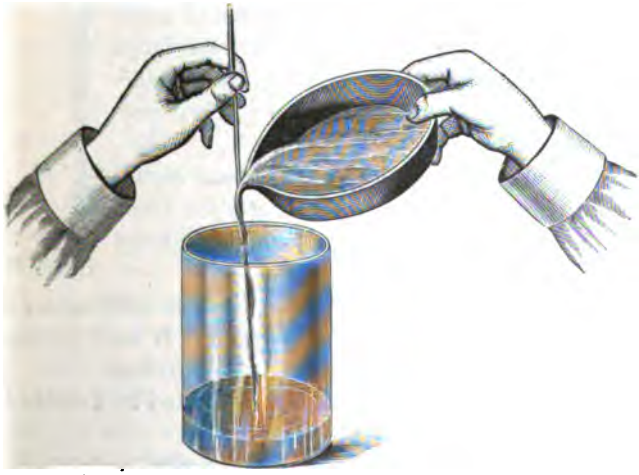


Fig. 22. Decantation by means of the "guiding rod."

steady hand. The rim or edge over which the liquid is to pass may be greased to obviate capillary attraction and thus prevent the liquid from running down along the outside of the vessel.

Another plan is to use a "guiding rod" to give the stream of liquid the proper direction.

98. The transfer of liquids from one vessel to another is also effected in various other ways, as by means of casseroles, pitchers, pipettes, syphons, etc.

Casseroles are dippers with lips and handles (fig. 23). They are generally made of porcelain, and are used principally for transferring considerable quantities of liquids, in portions, from one vessel to another in the course of laboratory processes for the production of chemicals.

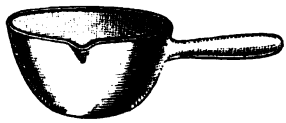


Fig. 23. Porcelain casserole.

Pipettes (fig. 24) are used for transferring small quantities of liquids. They are usually of glass and graduated.

99. Syphons and their use. Syphons of glass and of rubber tubing are indispensable, and are used for transferring liquids from vessels which can not or should not be moved or disturbed. Clear liquids may be almost completely withdrawn from heavy precipitates and sediments by means of the syphon.

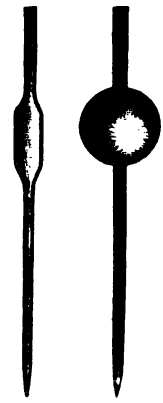


Fig. 24. Pipettes.

Syphons of glass, porcelain or earthenware are also employed for transferring solutions of acids or alkalies and other corrosive liquids from large containers, and for controlling the gradual addition of one liquid to another in processes of precipitation, oxidation, etc.

A variety of glass syphons will be found useful in any large pharmaceutical laboratory. Their construction and operation are shown in figs. 25 to 27.

Flexible pure rubber tubing of from 10 to 20 millimeters diameter is extremely convenient for syphoning. It may be required in lengths varying from 600 millimeters to 2 meters.

Two straight pieces of glass tubing connected by a rubber tubing joint make a good syphon.

Syphons work on the principle that "liquids seek their own level" through the force of gravitation. The syphon is a tube bent so that its two limbs would, if straight, meet at an acute angle. When about to be used, the instrument is filled with the liquid which is to flow through it; one limb is then inserted in the vessel

from which the liquid is to be withdrawn, and the other limb in the receiving vessel. The greater weight of the liquid in the longer limb of the syphon will cause it (the liquid) to flow down-



Fig. 25. Simple syphon.

ward, and the atmospheric pressure causes the ascent of fresh liquid through the shorter limb so that the flow is uninterrupted until the level of the liquid in both vessels is the same. When the level of the two bodies of liquid connected by the syphon is the same it is evident that the weight of the column of liquid in one limb is the same as the weight of the liquid in the other limb, equilibrium is established, and the flow ceases; if now one of the two vessels be lowered the flow of liquid through the syphon will begin again in the direction of the lower vessel.

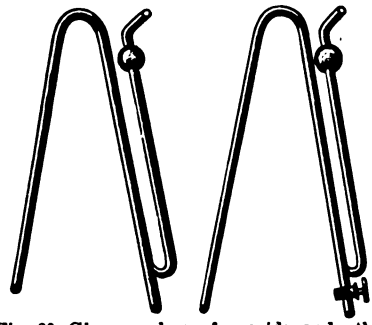


Fig. 26. Glass syphons for acids and other corrosive liquids.

100. Colation. Filtration through comparatively coarse media is called colation, and the liquid partially freed from solid particles by colation or *straining* is called the colature. Mixtures of liquid and solid matter are strained through sieves and through various kinds of *strainers*.

Unbleached muslin is the most generally useful straining cloth for ordinary laboratory operations in the production of inorganic

chemicals. The cotton cloth should be boiled in water before it is used in order to remove starch or other "sizing" and "filling" material. It should be cut into square pieces of various sizes to suit, and always larger than the frame upon which it is to be fastened for use. The most useful size of cloth strainer for the manufacturing laboratory is perhaps 600 millimeters square, and the stand required to hold such a strainer should be about 450 millimeters square and about 400 millimeters high, so that a stone jar of about 15 liters capacity may be placed under it. The strainer must be larger than the frame in order that a sufficient margin may be left for fastening the cloth on the wooden frame and for the necessary "bagging" of the strainer in addition to the allowance which must be made for shrinking.



Fig. 27. Arrangement for continuous filtration by means of syphons.

The straining cloth frame should be strong enough to stand constant use, and must be provided with strong pointed nails above on which to fasten the strainer. Square frames on four legs are much better than circular or triangular frames on three legs. In addition to the strainer stands, or frames on legs, an assortment of square frames without legs is also useful. Such a strainer frame is called a "tenaculum." (Fig. 29.)

Whenever the strainer is to be used for straining an aqueous fluid it should first be wetted in order to diminish the diameter of

the openings in the cloth by the swelling of the threads of which it is woven, and to establish uniform capillary attraction.

When large quantities of very heavy precipitates are to be washed or collected on a cloth strainer, care should be taken to make the strainer strong enough to bear the weight of the precipitate and the water. If a single thickness of the muslin is not strong enough, it is necessary to double it or to support the sag by broad muslin bands crossing each other under it, unless stronger muslin is at hand which is open enough to be suitable.

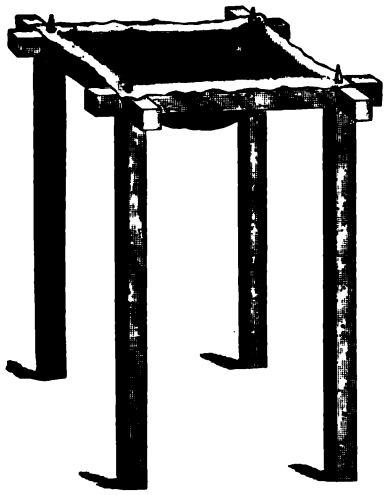


Fig. 28. Muslin strainer on stand.

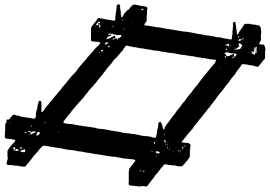


Fig. 29. Tenaculum.

Closely woven cotton cloth is not as suitable as a looser fabric. Linen is rarely used for strainers, but flannel is sometimes more



Fig. 30 and 31. Method of folding and twisting a small strainer.

effective than muslin, and seamless felt-bags are occasionally still more effective, as in the filtration of syrups and of liquids con-

taining undissolved particles of volatile oil or other liquid substances in suspension.

Pointed straining bags are also made of muslin and of flannel, and of "Canton flannel," as well as of felt.

The manner in which a strainer is folded and twisted to squeeze the liquid through, after it has ceased to run of its own weight, is shown in figs. 30 and 31. When the quantity of wet mass in the strainer is small in proportion to the size of the cloth, and the cloth not too large, it may be sufficient to twist the folded strainer with the hands. But when larger quantities are operated upon a *press* is necessary (par. 101).

In most cases, however, it is most advantageous to allow the wet mass to remain on the strainer until completely drained and sufficiently free from moisture to be spread out to dry, if the solid matter be the product and the liquid valueless.

Quantities of liquid exceeding two liters are usually filtered on strainers, lined with paper pulp if necessary, while smaller quantities should be filtered on funnels.

Liquids filtered through cloth strainers, and even through paper filters, do not always pass through quite clear from the start; they should then be returned to the filter until the filtrate runs clear.

To hasten the process the filter or strainer may constantly be kept nearly full until all of the liquid to be filtered has been added. But the filtration can not be hastened by scraping the sides of the filter or strainer if a clear filtrate is desired. Whenever practicable (as in a large laboratory) the straining stands used for washing precipitates should stand on an asphalt floor inclined toward one or more gutters connected with the drain pipes through which the valueless washings may pass away.

101. Presses. To express the liquid from voluminous magmas or wet precipitates holding large quantities of water a strong screw press is necessary. A practical, serviceable press must not be too small, because satisfactory results can not be obtained when the quantities operated upon are so great as to tax the capacity of the press to hold them. This is especially true of presses constructed like the ordinary pharmaceutical tincture press in which the solid matter subjected to the pressure is confined in a cylinder.

The mass of wet solid matter must be placed in the center of a *strong* press cloth, which is then folded so as to securely enclose the mass, and the size of the package must not be so large as to

cover the whole botton of the press within the cylinder, but should be of smaller diameter than that of the press block, so that plenty of room will be left around the bundle for the escape of the liquid expressed from it. Most of the failures in operating simple presses are due to the choking occasioned by trying to work on larger quantities of material than can be advantageously handled at one time. Small presses of brittle cast iron with press blocks of the same material and cylinders of tinned or galvanized sheet iron are, therefore, useless. Presses should instead be made of cast steel, or of hard wood, or of strong porcelain.

The pressure should be applied slowly or gradually. After a



Fig. 32. Witt's press, with press block of strong porcelain.

twist or two of the screw the liquid should be permitted to run until it stops before the screw is turned again. The final pressure should be strong.

When no more liquid can be squeezed out of the solid material in the press cloth the screw should be turned back; the package should be removed and the cloth unfolded, after which the contents may, if necessary, be broken up and mixed and again enfolded in the cloth, replaced in the press and subjected to expression a second time. A second treatment in the press is, however, rarely necessary in working upon inorganic substances.

Several serviceable presses are pictured here, two with one screw and one with two. One of the best is Witt's press. Hydrostatic presses and filter presses required for operating upon very large quantities of material will be found described in larger works on chemical technology and in the illustrated price catalogues of the makers of such apparatus.

Cylinder presses of from five to ten liters capacity are probably the most useful in pharmaceutical laboratories.



Fig. 33. Cylinder press.

102. Corrosive liquids may be filtered through various filtering media not affected by them. Acids, alkali solutions, solutions of zinc chloride and other corrosive solutions, when unclear, may generally be successfully clarified by subsidence and decantation or by drawing off the clear portion by means of glass syphons. But they may be filtered, when necessary, through washed sand, coarsely powdered glass or porcelain, glass wool, asbestos, powdered pumice stone, or powdered clay, according to circumstances.

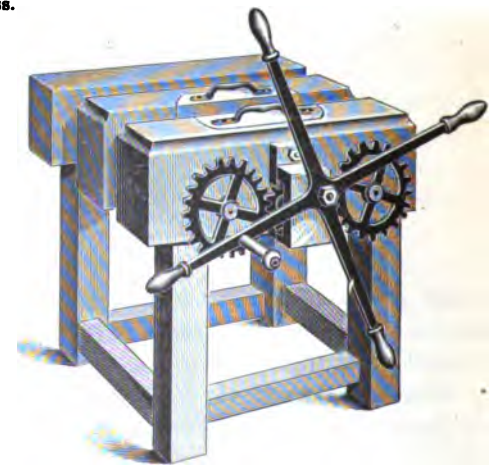


Fig. 34. Mohr's double screw press.

103. Paper filters. Filtration through paper is an extremely important and valuable method of clarifying liquids, and it is applicable to nearly all liquids which are not corrosive, or too viscous. Filter paper is unsized, porous paper, made expressly for the special use its name implies.

Gray filter paper is made of inferior material and should never

be used for pharmaceutical or chemical purposes because it may not only contain iron and other objectionable inorganic impurities, but is more liable to be infected with bacteria than white paper.

White filter paper is made of several grades of purity, thickness and porosity. A chemically pure filter paper leaving scarcely any ash on ignition is required for some purposes in quantitative chemical analysis; but for other chemical and pharmaceutical work it is only necessary that the white filter paper shall be free from iron and other impurities soluble in or affecting the purity of the liquids which may be filtered through it. The paper should be so pure and clean that even *hot* solutions of salts (except such salts as may chemically attack the cellulose of which the paper consists) may be filtered through it without the slightest discoloration and without taking up even traces of any substance from it.

Filter paper of very close texture (less porous paper) is required for the removal of extremely fine particles of solid matter; filter paper of loose texture, or more porous paper, is sufficient for the removal of coarser particles, and is necessary for rapid filtration.

Lighter paper is suitable for thin and light liquids, such as alcohol, etc.; heavier and coarser paper is preferred for the rapid filtration of water-solutions of inorganic substances, especially if the solutions be heavy; still coarser or more porous paper is necessary for the filtration of thick liquids such as syrups, oils, etc.

"Hardened paper filters" are best for the collection and washing of precipitates and for "pressure filtration."

Filter paper is sold in rectangular sheets as well as in circular form. Circular filters are made to fit the ordinary sizes of filter funnels. Whole sheets of filter paper may be used for cutting round filter paper; but this is, of course, a wasteful practice. Sheets of bibulous white filter paper are used mostly for absorbing water from precipitates, masses of small crystals, and other wet products.

104. When paper filters are used for collecting and washing precipitates, they should be *plain filters* which fit the funnel snugly so as to come in contact with and be supported by the inner sides of the funnel at all points in order that the precipitate may not

be spread upon a larger area of the paper than necessary, for if finely divided precipitates come in contact with a greater surface some loss must be occasioned thereby.

Plain filters are also used in *perforated funnels,* in *ribbed funnels,* and in *filter baskets;* all of which facilitate the passage of the liquid through the paper by providing openings and channels through which it may readily escape.

As a "plain filter" when opened forms a cone, the apex of which has an angle of 60 degrees, it follows that the funnel used for such a filter must be of the same angle.



Fig. 35. Corrugated funnel for rapid filtration.

But when a plain filter is used in a plain funnel of 60 degrees angle, the liquid can pass through the paper only at the apex of the filter in the throat of the funnel where the paper is not in contact with it. This would make the escape of the liquid very slow, which is a decided disadvantage except in the washing of precipitates. Moreover, the whole weight of the superincumbent liquid resting upon the paper at the apex of the filter, when the liquid is a heavy one and the filter full, may cause the paper to break at that point. To prevent this trouble, which is sometimes serious, the paper filter may be supported at its apex by an additional cone or filter of paper (or of perforated platinum) or of muslin. A paper filter "shod" with a muslin tip placed in the throat of the funnel is to be preferred to a "double filter" or a paper-shod filter.



Fig. 36. Perforated porcelain funnel for filtration.



Fig. 37. Perforated porcelain funnel for draining crystals or for filtration.

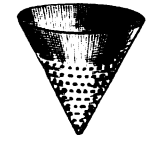


Fig. 38. Perforated platinum cone for filtration by pressure.

Plaited filters are the most useful paper filters for rapid filtration when plain funnels are used without filter baskets.

105. How to fold paper filters. A circular piece of filter paper may be folded into a plain paper filter by making the first fold

a straight line through the center, the crease thus made being coincident with the diameter of the circle; the second fold is then made through the center of the first fold and at right angles with it; a third fold is next made, again dividing the folded paper into two equal segments of the circle, the crease running from the apex to the edge. The folded paper is now opened up until the half-circle is reached, after which the flaps are laid back on opposite sides against the second fold, and the two center edges of the filter are then parted so that the cone thus formed presents three thicknesses of paper on two opposite sides with a single thickness of paper on the two other opposites, as shown in Fig. 39.

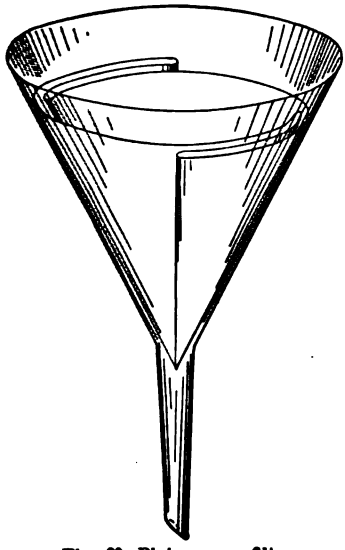


Fig. 39. Plain paper-filter.

A plain filter may be made out of one half-circle of filter paper, as shown in Fig. 40, and this kind of paper filter may be made to fit any funnel, since the fold can easily be made broader either at the base or at the apex of the cone so as to give any angle desired. This filter, as will be seen, requires the use of only half

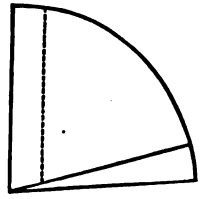


Fig. 40. Simple filter made of one half-circle of paper.



Fig. 41. Plaited paper filter.

as much paper, which makes it the most economical paper filter that can be made.

The *plaited filter* can be made in various ways. The most common method is sufficiently illustrated by fig. 41.

Plaited filters may be used with funnels of any angle.

106. Double, treble and even quadruple paper filters and cloth strainers are sometimes used when a single thickness of the paper or cloth is insufficient to produce a clear filtrate. But some turbid liquids holding extremely finely divided solid matter in suspension, or containing some insoluble liquid distributed through it with which it forms an emulsion, can not be rendered clear by any process of filtration. The pores of the paper may be gradually entirely closed by finely divided precipitates, very viscous fluids (like strong mucilages, strong solutions of albumin, etc.) do not pass through filter paper at all, and "emulsions" of mixed liquids pass through without separation if at all.

107. Hot filtration. Many liquids which, when cold, refuse to pass through the ordinary filtering media, including paper, may be successfully filtered when hot. Again, many liquids which pass through the filter slowly when cold, may pass less slowly or even rapidly when hot.



Fig. 42. Funnel in hot-water-jacket for hot filtration.



Fig. 43. Arrangement for hot filtration.

Jacketed funnels (Figs. 42 to 44) are employed for hot filtration, and also conical coils (Fig. 45).

108. Rapid filtration is, in special cases, effected by hydrostatic pressure or by suction.

One good contrivance is a box having perforated sides and covered on the outside with the straining cloth (or filter paper next to the box and cloth over it) securely fastened, this box to be provided with a tube through which a syphon can be inserted by

means of which the filtrate accumulating in the box can be drawn off. This box is then submerged in the turbid liquid to be filtered, when the hydrostatic pressure causes the liquid to pass through the cloth (and paper) into the box.



Fig. 44. Dietrich's jacketed funnel for hot filtration; used with steam heat.



Fig. 45. Hot-water-coil for hot filtration.

Various kinds of "filter pumps" are also used to create a partial vacuum in the receiving vessel into which the filtrate

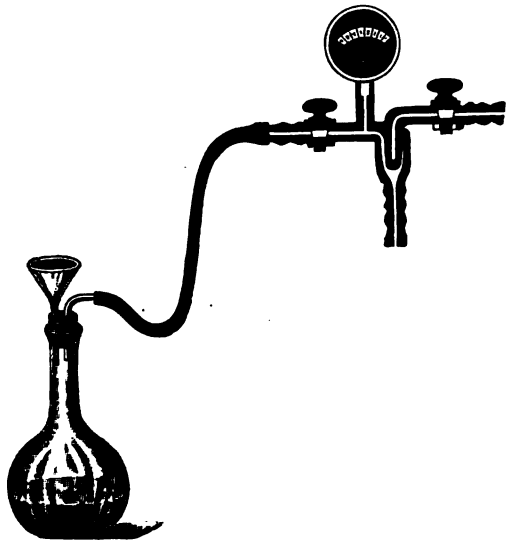


Fig. 46. Filtration under pressure by means of a filter pump which exhausts the air from the flask.

passes so as to cause suction through the filter. One such filter pump is shown in Fig. 46.

A perforated filtering disc of porcelain with a rubber tire around its grooved edge, is placed in the funnel and covered with filter paper, muslin, or both, or flannel, as may be required. The funnel is fitted tightly into the neck of a strong receiving bottle (Fig. 47) and this is connected by a side-tube with the filter pump. (See also Fig. 19.)



Fig. 47. Use of perforated discs for filtration under pressure.



Fig. 48. Arrangement for continuous filtration.



Fig. 49. Illustrates an arrangement for the automatic flow of liquid in filtration.

109. Continuous filtration. Figures 48 and 49 illustrate a simple arrangement for a continuous flow of liquid into the funnel for filtration or for washing precipitates.

Continuous filtration may also be effected by a syphon arrangement as shown in Fig. 27.

CHAPTER VII.

EVAPORATION.

110. The temperature at which any substance assumes the state of vapor depends upon its constitution and upon the pressure to which it may be subjected. Different substances exhibit greater or less differences as to their vaporizability according to their composition.

Many substances remain solid at the highest temperatures producible; others may be liquefied but not vaporized; others decompose before they undergo any change of their state of cohesion or aggregation; many kinds of compound matter exist only in the gaseous state under ordinary conditions of temperature and pressure; but numerous compounds which are solid or liquid under ordinary conditions can be more or less readily converted into vapor by sufficient heat, or by reducing the pressure to which they are exposed, or by both of these means.

The power with which gases and vapors resist compression into the liquid or solid state is called their *vapor tension.*

But the conversion of solids and liquids into vapors depends not upon composition, temperature and pressure, only; it may be greatly affected by various forms of molecular attraction. Heat opposes molecular attraction and therefore breaks down cohesion; pressure, on the contrary, aids cohesion. But the molecular attraction between different kinds of matter may oppose and in many cases overcome the cohesion between like molecules. Thus water vapor passes off from ice even at the freezing point if the air surrounding the ice is *dry*—i. e., if the avidity of the air for water is not already satisfied.

111. Boiling point. The boiling point of any liquid is the temperature beyond which it can not continue in the liquid state of cohesion without the aid of increased pressure.

The boiling point of any given liquid is constant under constant pressure.

Carbon dioxide boils at —42°.44 C.; ether at 37°; alcohol at 78°; water at 100°; mercury at 357°; and zinc at 940°. These

boiling points refer to the ordinary atmospheric pressure; they rise as the pressure is increased and fall with diminished pressure.

The boiling point of any liquid is, in other words, the temperature at which the tension of its vapor is greater than the pressure which it sustains.

112. Evaporation is the formation of vapor at or from the surface of any solid or liquid at any temperature below its boiling point.

When comparatively rapid evaporation is desired it is aided by the application of heat, by the diminution of pressure, and by other means.

When very slow evaporation is desired, as is frequently the case in the production of crystallized salts, no heat is applied. Evaporation without the application of heat is called *spontaneous evaporation.*

Evaporation depends largely upon molecular attraction between the molecules of the vapor and the molecules of the components of the superimposed air. This attraction is closely akin to that which causes solution.

113. Vaporization, as this term is generally used, means the formation of vapor at the boiling point, and it differs from evaporation chiefly in the fact that the vapor which is formed at the boiling point of any liquid is not formed at or from the surface of the liquid, but in the body of it, and mainly at the surface of contact between the liquid and that portion of the vessel containing it, which is directly exposed to the source of heat.

114. The **rate of evaporation or vaporization** of any liquid depends upon various conditions, among which the following are the most important:

1. *The volatility of the liquid,* which depends upon its constitution or composition. Thin, mobile, less cohesive liquids are more volatile, even if of greater specific weight, than thick, cohesive liquids; but the specific resistance of any liquid to its conversion into vapor, or, in other words, the relative force of attraction between its molecules, must depend primarily upon its chemical structure.

2. The *temperature which the liquid attains* has a direct and decided influence upon the rate of evaporation. A boiling liquid

remains of constant temperature, but the rate of evaporation below the boiling point is greater the higher the temperature.

3. The *supply of heat* for the formation of the vapor has an important bearing, because the quantity of heat motion necessary for that purpose is a fixed quantity for each substance. Whenever any given liquid passes into vapor it takes up a definite quantity of heat which is, therefore, called the latent heat of its vapor. The latent heat of any given kind of vapor is a constant quantity; it does the work of keeping the substance in the state of vapor, is the cause of its vapor tension, and is liberated whenever the vapor is condensed.

The rapid or free application of high heat must, therefore, favor a rapid rate of vaporization.

In this connection we must consider the thermal conductivity of the vessel in which the liquid is heated, and also the extent of surface of that vessel with which the flame or other source or means of application of the heat comes in direct contact.

Shallow vessels are more effective than deep ones if only the bottom of the vessel is exposed to the heat applied. Silver dishes are more effective than any other because silver is the best conductor of heat known.

4. The *pressure* to which the liquid is subjected reduces the rate of evaporation by resistance to the vapor tension. Heavy liquids, other conditions being equal, do not evaporate as rapidly as lighter liquids.

Solutions and mixtures which acquire increased density by the concentration due to the evaporation of the solvent, or of one of its more volatile constituents, do not have constant boiling points, except saturated solutions. When a dilute aqueous salt solution is boiled its boiling point rises as the solution becomes more and more concentrated and does not become stationary until a saturated solution is formed. The higher boiling point is here caused by increased pressure.

Deep vessels are unfavorable to rapid vaporization if filled or nearly so for the vapor is formed mainly at the bottom, and if the depth of the liquid is great the vapor must be formed under greater pressure. The release of the vapor may, however, be facilitated by stirring.

5. The rate of evaporation or vaporization is, finally, aided by *molecular attraction between the vapor formed and the gases*

of which the air consists. The proportional extent of surface of the evaporating or boiling liquid which comes in contact with the air is, therefore, a matter of importance, not only because greater depth causes increased pressure upon the lower strata of the liquid, but also because actual contact is necessary to the diffusion of the vapors into the air. This is an additional reason for preferring shallow vessels rather than deeper ones.

The surface of contact between the air and the liquid may be increased by stirring the latter.

The humidity of the air in contact with the liquid also has its effect upon the rate of evaporation of water, for the power of the air to absorb the vapor is not unlimited.

The rate of evaporation or vaporization is greatest when the air is free from vapor of the same kind as that being formed, and when the air into which the vapor passes becomes saturated with it, no further aid is to be derived from the contact of the liquid with that air. Currents of warm, dry air over the surface of the liquid greatly hasten the formation of vapor, and such currents are created by stirring the liquid as well as by other simple means, whereby the saturated air is removed and replaced by a fresh supply capable of taking up the vapor.

115. The **objects** of evaporation and vaporization are: 1, to concentrate solutions by the elimination of a part of the solvent; 2, to obtain saturated solutions for the purpose of causing crystallization; 3, the completely separate volatile liquids from solutions and mixtures.

Liquids are evaporated: 1, to dryness; 2, to a given volume; 3, to a given weight; 4, to a given density; 5, to a certain more or less definite consistence; 6, to the point at which solid matter begins to separate; 7, to a constant weight (i. e., until there is no further loss of weight); or 8, until a certain objectionable volatile constituent or impurity shall have been completely expelled.

116. Boiling-vessels should be deep if rapid vaporization is to be avoided as far as practicable—i. e., when the object is the maintenance of the boiling point purely for its chemical effects. Deep dishes, dishes covered by inverted funnels or by other covers, flasks, beakers and various other cylindrical vessels—all these are good boiling-vessels, but poor evaporating-vessels.

117 Evaporation-vessels. The shallow dishes, kettles, or pans employed for the vaporization or evaporation of liquids

should be made of materials which are sufficiently good conductors of heat, not affected by the substances with which they must come in contact, and strong enough to resist fracture.

The most common materials are porcelain, other earthenware, glass, iron, tinned iron, pure tin, copper, tinned copper, enameled iron, silver, platinum and aluminum.

Silver dishes are very desirable in cases in which high temperatures are safe and the metal not attacked by the liquids heated.

Porcelain dishes which can bear sudden changes of temperature without danger of fracture are the most generally useful evaporating dishes.

But hot glass and porcelain vessels should never be put on cold or wet surfaces; it is better to let them cool gradually, or to put them upon rings of straw, or grommets of rubber tubing, or upon dry cloths, tow or cotton.



Fig. 50. Round-bottomed evaporation dish of porcelain.



Fig. 51. Flat-bottomed evaporation dish of porcelain.

Glass and porcelain vessels should not be heated too suddenly, and should be dry on the outside when dry heat, as the bare flame, is applied to them. Wire gauze, or wire cloth, or a sand bath, or other baths, may be interposed between the flame and the glass or porcelain, so as to distribute the heat evenly over the whole bottom of the vessel. The flame should also be well regulated so as to be not unnecessarily high, and so that the heat may be applied gradually.

A complete assortment of sizes of porcelain evaporating dishes is necessary to every well-equipped pharmaceutical laboratory for the production of chemicals—from 50 millimeters to 400 or 500 millimeters diameter.

Metal vessels, except silver, are rarely useful for chemical work, and enameled iron (agateware, etc.) is not reliable as the enamel is too liable to crack off or to become perforated by "pin holes."

118. Modes of application of heat for the evaporation or vaporization of liquids. According to the temperature required the heat may be applied: 1, by direct flame; 2, with but a piece of wire cloth interposed between the flame and the vessel; 3, by the sand bath; 4, through asbestos cloth; 5, by the glycerin-bath, or an oil-bath, or various salt solution baths; 6, by direct steam; 7, by steam jackets; 8, by steam coils in or around the vessel; 9, by the water-bath; or 10, by a hot air bath.

The student is referred to the chapter on heating apparatus for further information on this subject.

119. Dangers of over-heating in the evaporation of solutions. The temperature can generally be easily controlled by means of baths, with the aid of thermeters, by the regulation of the flame or fire, by gradual elevation of the temperature, and by watching the operation. Should there be any signs of danger, prompt removal of the flame or heater, or of the vessel from the source of heat, is necessary.

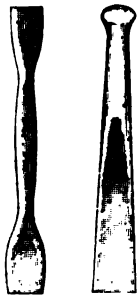


Fig. 52. Porcelain stirrers.

Vacuum-pans are much employed in the evaporation of solutions of organic substances, but not in the operations of inorganic pharmaceutical chemistry.

When water-solutions are evaporated very high heat, up to the boiling point, may be safely applied in many cases so long as the solutions are dilute, but it must be remembered that as the density of the solution increases the boiling point also rises, and the danger point may be reached unless the thermometer is used as a guide.

If solid matter separates from the liquid during the process of evaporation, constant stirring is generally necessary to prevent accidents; should the solid matter form a pellicle over the liquid, the temperature might then rise too high, and if the solid matter deposits on the bottom of the dish the latter may crack or the deposited matter may become overheated.

The stirrers used may be of glass, porcelain, wood, or other suitable material. They may be in the shape of rods, spatulas, spoons, or ladles.

120. Slow evaporation may be performed over a well regulated steam-bath, water-bath, sand-bath, or air-bath, in the drying closet, or by spontaneous evaporation without any application of heat.

The evaporating dish should be loosely covered with paper, or should be placed under a hood to protect the contents from the dust.

121. When objectionable vapors pass off from evaporating liquids the employment of hoods or fume chambers is necessary unless it be found practicable to carry on the operation out of doors, in which case the operator may easily avoid the gases by standing with his back to the quarter from which the wind comes.

122. Expulsion of hygroscopic moisture. Small amounts of water held by hygroscopic substances which can not safely be exposed to heat may be removed by means of **desiccators** which consist of glass covers placed over vessels containing calcium chloride, sulphuric acid, dry potassium hydroxide, dry lime, or other substances having a great avidity for water. The dishes, beakers, watch-crystals, or other vessels containing the substance or solution to be subjected to *desiccation* are placed immediately over, or below, or beside the vessel containing the calcium chloride or other substance employed to absorb the water as it evaporates.



Fig. 53. Desiccating apparatus.

CHAPTER VIII.

DISTILLATION.

123. Distillation is the vaporization of a liquid in an apparatus so constructed that the vapor which is formed in one vessel is conducted into another vessel in which it is condensed back to the liquid state and collected.

The object of distillation is the separation of volatile liquids from non-volatile substances with which they are mixed, or which they contain in solution; or the separation of more volatile from less volatile liquids.

The liquids subjected to distillation may be mixtures or solutions consisting of two or more liquids; or solutions of substances which are solids when separated from the solvent; or mixtures of solid and liquid substances not forming solutions; or liquids containing comparatively small proportions of other substances which it is desired to separate.

124. Many different substances have different degrees of volatility within the range of temperatures produced in ordinary distillation, while other substances are quite non-volatile at those temperatures.

The volatile liquid carried over by the distillation forms the product called the *distillate.* The less volatile or non-volatile matter left in the vessel in which the vaporization was effected is called the *residue.* This residue may be either liquid or solid.

The separation of a volatile liquid from an altogether non-volatile substance with which it may be associated by solution or otherwise is easily effected by distillation.

But the separation of two or several more or less volatile liquids from each other by distillation is difficult, and scarcely possible unless their respective boiling points differ sufficiently. Such a separation is called *fractional distillation.*

125. The *latent heat of vapor* is the quantity of heat required to hold it in its gaseous state. It is called "latent" because it does not register upon the thermometer or reveal itself in any other way than by performing the work of keeping the molecules of the volatile substance apart from each other so as to hold them in

the condition constituting what is known as "vapor," and the energy required to do that work can not at the same time do any other work.

The latent heat of water vapor or steam is about 537° C., for the amount of heat energy which is necessary to convert 1 Gram of water into vapor (or to evaporate 1 Gm of water) is precisely the same as the heat energy required to raise the temperature of 537 Grams of water one degree (centigrade).

The latent heat of alcohol vapor is expressed by about 375°; and that of ether by about 163° C.

The quantity of heat energy required to raise the temperature of a given quantity of water from 0° to 100° C. is a constant quantity, and the quantity of heat energy required to convert water of 100° into vapor, and to hold it in a state of vapor, is 5.37 times as great as the quantity of heat energy required to raise the temperature of the same amount of water 100°. The quantity of heat energy, or thermal energy, is measured in units which stand for the amount of heat required to raise the temperature of one liter of water one degree (centigrade). The quantity of heat energy required to raise the temperature of one liter of water from 0° to 100° C. is, therefore, 100 heat units, or thermal units. But the quantity of heat energy required to convert one liter of water at 100° into vapor, and keep it in a state of vapor, is 537 thermal units. Hence 637 units of thermal energy will be required to convert one liter of 0° into water vapor of 100°.

Whenever the vapor produced by one liter of water is condensed or converted from vapor at 100° into liquid water at the same temperature, the latent heat of that vapor, amounting to 537 units, is released.

126. The most common method of causing the condensation of vapor in distillation is to conduct the vapor through tubes surrounded by water of the ordinary temperature (between 15° and 20° C.). This water is called the *condensing-water,* and it serves the important purpose of absorbing the heat given up by the vapor. The vapor can not be condensed unless its latent heat be transferred to or absorbed by the substances with which it comes in contact; but the rate at which condensation takes place is in direct proportion to the rate of absorption of the latent heat of the vapor by the vessel in which it is condensed, by the water surrounding that vessel, and by the contiguous air.

The greater part of the heat lost by the vapor is taken up by the condensing water. The heat lost in cooling is precisely the same amount as is required to raise the same body through the same number of degrees. If one kilogram of water at 10° be mixed with the same quantity of water at 90°, the equalization of temperature would result in two kilograms of water of 50°, for the amount of heat gained by the water of 10° would be one-half of the difference between 10° and 90°, and the heat lost by the water of 90° would be the other half of that difference.

If it be assumed that all of the latent heat of the steam in the distillation of water is transferred to the condensing water, and that all of the condensing water enters the condenser at a temperature of 15° and is heated to 70°, running off at that temperature, and if it be further assumed that the distillate passes out having a temperature of 80°, then, as the latent heat of water vapor is 537 thermal units, it follows that about 10.74 liters of condensing water will be required to take up the latent heat given up in the formation of one liter of distilled water, and nearly 0.25 liter of additional water of 15° will be required to reduce the temperature of that distillate from 100° to 80°, so that about 11 liters of water of 15° would have to be supplied to the condenser in making each liter of distilled water of 80°. But in actual practice the amount of condensing water required is about twice as great, for it is impossible to regulate the operation and prevent waste. All that can be done in this direction is to see that the condensing water supplied runs off nearly as hot as the distillate itself.

127. Boiling points of mixed liquids. When a mixture of several liquids of different boiling points is heated, it boils at a temperature somewhat higher than the boiling point of the most volatile constituent of the mixture.

128. Use of **thermometers** in distillation. In order to regulate the temperature in distillation, whenever necessary, the mercury thermometer is often brought into requisition. The slender special laboratory thermometer of glass used for this purpose is represented by Fig. 54. It is long, of very fine bore, and has a very small bulb. Such thermometers range from about —35° to +200 C., but for ordinary purposes the most useful are those which register temperatures from —20° to 100°. Well made

laboratory thermometers do not break easily, are not easily fractured by heat, and may be conveniently inserted in stills, flasks, retorts, and other vessels by means of perforated rubber stoppers or corks.

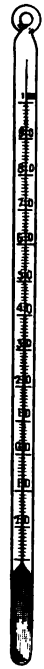


Fig. 54. Laboratory thermometer.

129. *Simple distillation* is exemplified in the production of distilled water, which is fully described elsewhere in this book.

Chemical distillation is the production of a volatile liquid by chemical reaction and the separation of that product from the bye-product by distillation, the reaction being effected in the distilling apparatus.

130. Distilling apparatus. Large *stills* are made of copper, iron, tinned iron, lead or stoneware. Small stills, having a capacity of from 5 to 100 liters, may well be made of the same materials.

The best stills are usually those of the most simple construction, which can be readily cleaned and are not liable to get out of order.

Glass flasks and retorts are used for small operations. Porcelain retorts or stills are also occasionally used.

Distilling flasks must be of the best Bohemian glass, of good shape, thin, not of uneven thickness, well annealed. The necks of distilling flasks are sometimes required to be rather long, but more frequently short.

A *retort* is practically a flask with a long neck bent at an angle of about eighty degrees to the body. The most useful glass retorts are those having one or two *tubulures* or necks with ground glass stoppers.

The distilling apparatus also includes a *condenser* and a *receiver.* It is often practicable to combine condenser and receiver, for when comparatively small amounts of liquid are distilled and the product is not too volatile the receiver may also serve as the condenser.

Glass receivers are usually globe-shaped, with wide necks, as shown in Fig. 55. When fitted to retorts without the intervention of condensers they may be placed in cold water or in broken ice, or a stream of cold water may be kept running upon them. The necks of retorts and receivers, or the tube connecting them, may be covered by a wet cloth kept cool by running water.

Retorts are always of awkward shape, and their liability to breakage lessens their usefulness. Flasks are to be preferred whenever practicable.



Fig. 55. Illustrating distillation on a small scale. The condenser is a Liebig condenser.

The *fittings* by which flasks or retorts are connected with condensers or receivers, or both, consist of bent glass tubing, T tubes, rubber tubing, soft sheet rubber, and perforated rubber stoppers or corks. The whole apparatus should be securely supported, the connections instead of being rigid should be somewhat elastic to diminish the danger of breakage, and all the joints should be tight.



Fig. 56. A tubulated glass retort.

The *cork-borer* is necessary when corks are used instead of rubber stoppers, and only the very best kind of cork can be used. Perforated rubber stoppers of pure (black) rubber of all ordinary sizes and with one, two, or more perforations for



Fig. 57. Glass fittings for distilling apparatus and for gas apparatus; elbow, T tube, and glass-stoppered piece of tubing.

the insertion of glass tube elbows or T tubes, safety-tubes, reflex condensers, and thermometers, any or all, can be obtained from any dealer in chemical apparatus and are so far superior to cork that they should altogether displace the latter. (Fig. 60.)

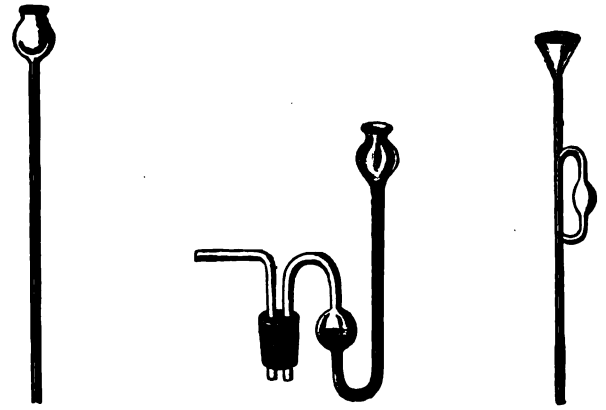


Fig. 58. Thistle-tube and safety tubes for distilling flasks and gas apparatus.



Fig. 59. Cork-borer of brass tubing.



Fig. 60. Perforated rubber stoppers.

131. The *worm condenser,* or "condensing worm," is a simple, common and effective condensing apparatus employed in connection with large stills, especially for the distillation of water or of

alcohol. It consists of a spiral block tin pipe placed in a tub, barrel or tank, through which cold water flows.

Liebig's condenser is shown in Fig. 55. It is constructed of two tubes which may be taken apart and cleaned. The vapor passes through and is condensed in the inner tube, which is surrounded by the cold water passing through the outer tube.

Squibb's condenser is a modification of Liebig's. Fig. 61.

Mitscherlisch's condenser is a double cylinder placed in a ves-

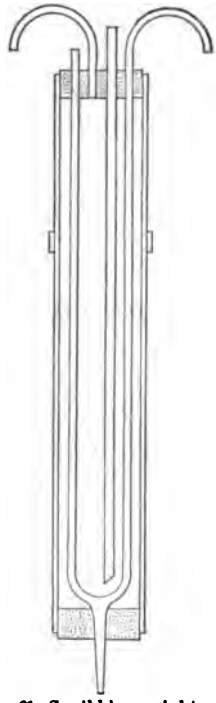


Fig. 61. Squibb's upright condenser.

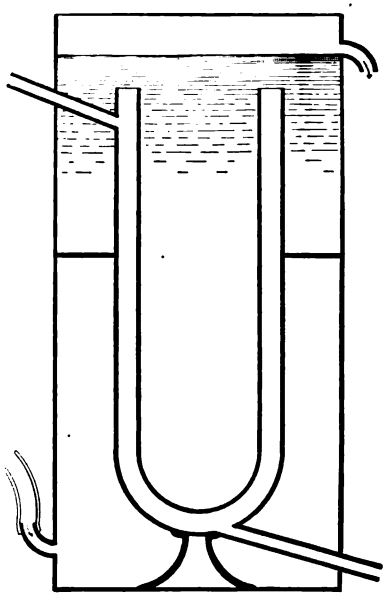


Fig. 62. Vertical section showing construction of Mitscherlisch's condenser.

sel of cold water. The double cylinder is made of two tubes tightly fitted together at both ends. The vapor is condensed *between the two tubes,* and the cold water passes around the outer tube and through the inner tube.

132. *Stands* for retorts, flasks, condensers, etc., are made of iron, or of iron and wood. They should be strong enough, and

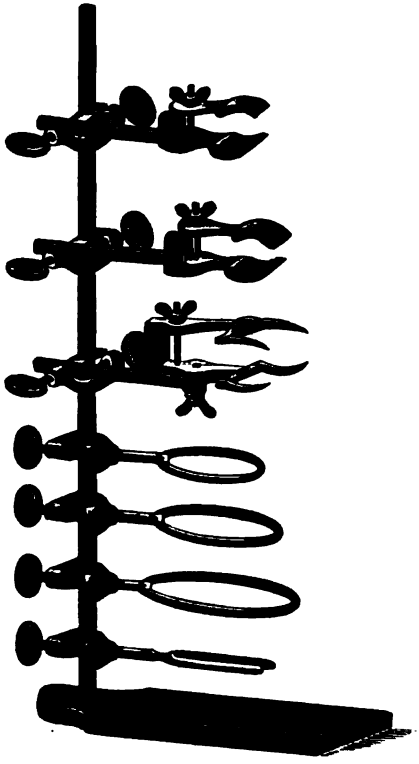


Fig. 63. Apparatus stand of iron for retorts, flasks, condensers, etc.

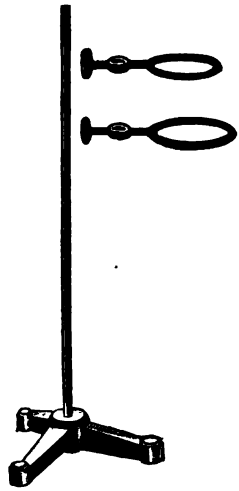


Fig. 64. Iron stand for funnels, flasks, beakers, etc.

provided with a sufficiently broad base, heavy, and with the center of gravity in that base.

CHAPTER IX.

CRYSTALS AND CRYSTALLIZATION.

133. A **crystal** is a naturally formed geometric solid bounded by smooth plane *faces* meeting each other to form straight *edges* and solid *angles*. The edges are formed by two contiguous intersecting faces; the solid angles or corners are formed by three or more faces intersecting each other at one point.

Calcspar, rock crystal, diamond, galena, alum, blue vitriol, green vitriol, Epsom salt, and quinine sulphate furnish examples of crystals.

134. **Crystallization** is the formation of crystals. But for our present purposes we shall use the term to signify the preparation of chemical products in the form of crystals.

Crystallizable substances are *crystallic* when they occur in comparatively well defined, free or detached individual crystals, such as we see in alum, copper sulphate, sodium carbonate, rock crystal, and potassium ferrocyanide; they are *crystalline* when their crystalline structure is evident throughout their mass, but the crystals are very small, imperfect, and not detached or separable from each other, as in masses of ferric chloride, black antimony sulphide, camphor and ammonium chloride.

Substances consisting of such minute crystals that they have the form of coarse or fine powder may be either crystallic or crystalline, but are always described as crystalline. Powders consisting of minute crystals are sometimes described as "crystal meal." When the crystals are so small that they can not be recognized without the aid of the microscope, the substance made up of such crystals is said to be *micro-crystalline.*

Substances which present no indications of crystalline structure are called *amorphous.*

135. Only molecules of the same kind form crystals, or arrange themselves into regular polyhedral solid bodies, except that water (and sometimes alcohol) of crystallization can be included. Hence when any substance crystallizes into well defined crystals

other substances which may be present with it are excluded, so that crystallization is a good indication of probable purity.

But crystallization is not absolute evidence of purity, for, although it is true in a general way that crystals are chemically homogeneous or consist of but one kind of molecules, it happens quite frequently that other kinds of molecules are *mechanically* included in the crystals formed, or that the exclusion of foreign substances is not complete until after repetition of the process of crystallization once, twice, or several times. Moreover, there are some substances which freely crystallize together and the crystals of which grow in each other's solutions.

136. There are innumerable distinctly different forms of crystals; but, as a rule, each particular kind of matter crystallizes in but one form, so that the crystal form of a substance is one of the means by which it may be identified.

Numerous different substances may, however, crystallize in the same form even if they can not crystallize together to enter into the formation of the same individual crystals. It happens, too, that *apparently* the same species of molecules may crystallize in two or three different forms.

137. Substances crystallizing in two different forms are called *dimorphous substances;* those crystallizing in three forms are *trimorphous* or *polymorphous.*

When different substances crystallize in the same form they are said to be *homoeomorphous.* But homœomorphous substances are frequently of altogether dissimilar internal structure.

Isomorphous substances have not only the same crystalline form but also perfectly analogous internal structure. They generally contain the same number of atoms in their respective molecules; the systems of interatomic linking of any two or more isomorphous substances are identical; they contain the same number of molecules of water of crystallization, if any; they crystallize together in the same crystals from a common solution; and their crystals may grow in each other's solutions (or the crystal of a less soluble substance may be made to grow in a more soluble isomorphous substance). Examples of isomorphous substances are: the alums (although an ammonium alum does not contain the same number of atoms as the corresponding alum formed by any alkali metal); the phosphates and arsenates of the alkali metals; the halides of the alkali metals; the nitrates of potassium,

rubidium and cæsium; the carbonates of magnesium and calcium; the sulphates of magnesium and zinc; etc.

Sodium nitrate, $NaNO_3$, and calcium carbonate, $CaCO_3$, crystallize in similar forms, and their molecules contain identical numbers of atoms; but they are not isomorphous because their systems of atomic linking are altogether dissimilar owing to the fact that sodium is a monad and calcium a dyad, and that the nitrogen of $NaNO_3$ is a pentad, while the carbon of the $CaCO_3$ is a tetrad—



Sodium nitrate and calcium carbonate are accordingly only homœomorphous.

Elements having different valences can not replace each other and form isomorphous substances.

Different substances which crystallize in different forms are *heteromorphous.* They never crystallize together in the same identical crystals.

When heteromorphous substances are contained together in the same solution they may be separated from each other by crystallization, especially if their respective solubilities differ sufficiently; but isomorphous substances are difficult to separate in this way, even if they do not freely enter into the formation of the same individual crystals.

Less soluble substances, of course, crystallize before the more soluble substances from the same solution.

But the presence of two or more substances in the same solution may cause one of these substances to form crystals having the form belonging to the other or one of the others. For instance, copper sulphate crystallizes normally in the triclinic form, but if it crystallizes from a solution containing about one-seventh as much ferrous sulphate as copper sulphate, the copper salt will crystallize in monoclinic prisms, the form of the crystals of ferrous sulphate.

138. *General descriptive terms,* referring to the forms of crystals, are so numerous that they can not be mastered without a more extended study of crystallography than is possible in this book.

The *axes* of crystals are the several directions of their extension. As geometric solids must have at least three directions of extension—length, breadth and thickness—it follows that no crystal can have less than three axes; but crystals of the hexagonal

CRYSTALLINE FORMS OF THE REGULAR SYSTEM.

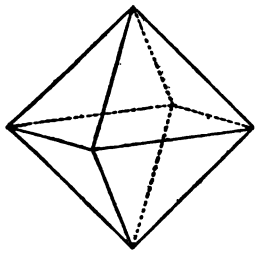


Fig. 65. Regular octohedron.

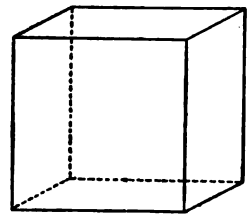


Fig. 66. Cube.

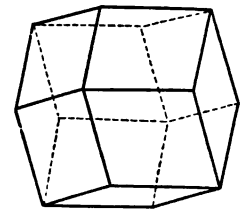


Fig. 67. Rhombic dodekahedron.

system have four axes. The forms of crystals depend primarily upon; 1, the number of the axes; 2, the angles at which the axes severally intersect each other, and 3, the respective lengths of the several axes with regard to each other. All the crystalline forms are grouped into six distinct *systems,* called:

CRYSTALLINE FORMS OF THE HEXAGONAL SYSTEM.

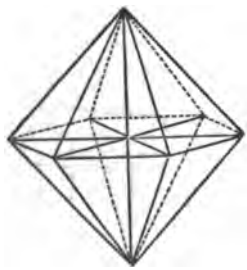


Fig. 68. Double six-sided pyramid.

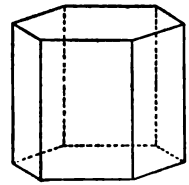


Fig. 69. Six-sided prism.

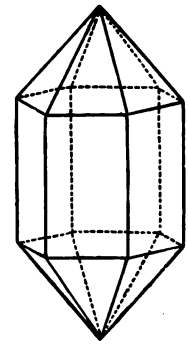


Fig. 70. Hexagonal pyramidal prism.

I. **The Regular System** (monometric, tessular, or cubic system).—Crystals having three axes of equal length intersecting each other at right angles. The facial angles or edges are, therefore, also right angles.

The fundamental forms of this system are the cube, the regular octohedron, and the rhombic dodekahedron.

II. **The Hexagonal System** (rhombohedral system).—Axes

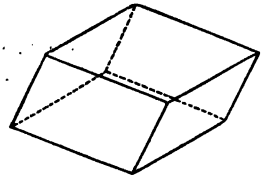


Fig. 71. Hemihedral rhombohedron.

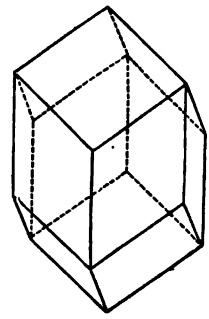


Fig. 72. Combination of rhombohedron and prism.

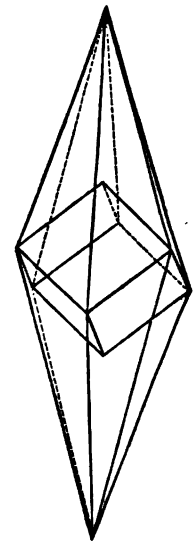


Fig. 73. Scalenohedron with inscribed hemihedral rhombohedron.

CRYSTALLINE FORMS OF THE QUADRATIC SYSTEM.

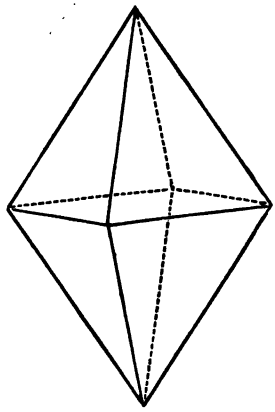


Fig. 74. Square-based double pyramid. (Octohedron.)

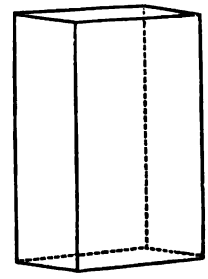


Fig. 75. Quadratic prism.

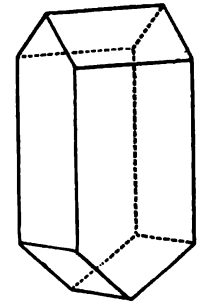


Fig. 76. Form found in crystals of the sulphates of magnesium and zinc.

four. Three of these axes are of equal length; these are called the *secondary axes*. The fourth, called the *primary axis*, is either longer or shorter than the other three. The secondary axes are all in the same plane, and cut one another at angles of 60 degrees; the primary axis is at right angles to the plane of the other three.

The fundamental form of this system is the double six-sided pyramid. Other important forms are the regular six-sided prism, and the rhombohedrons.

III. **The Quadratic System** (the dimetric, square, prismatic, pyramidal or tetragonal system).—Three axes. The two *secondary axes* are of equal length; the *primary axis* is longer or shorter. The axial angles are all right angles.

Pyramids of this system have square bases.

The dominant forms are the double four-sided, square-based pyramid and the right square prism.

IV. **The Rhombic System** (trimetric, or right prismatic system).—The three axes, all of unequal lengths, cut each other at right angles.

The fundamental form is the right rhombic double pyramid, or rhombic-based octohedron.

V. **The Monoclinic System** (monosymmetric or oblique prismatic system).—The three axes are of unequal length; the two *secondary axes* are at right angles to each other; the *primary axis* is at right angles to one of the secondary axes, but forms oblique angles with the other.

The primary form is the monoclinic pyramid.

VI. **The Triclinic System** (the assymmetric, or double oblique prismatic system).—The three axes all of unequal length, and the axial angles all oblique.

The fundamental form is the triclinic pyramid.

139. *Cubes* belong to the regular system.

Prisms are to be found in all except the regular system. Prisms with *rectangular* sides if they are six-sided belong to the hexagonal system; they belong to the quadratic system if four-sided and square-based. Prisms with oblique angles or rhomboid sides and bases belong to the monoclinic system if any two of their axes are at right angles; but to the triclinic system when they have no right axial angles. Prisms of the rhombic system have rectangular but not square bases.

Pyramids belong to all the six systems. Those with square bases belong to either the regular or to the quadratic system; if their faces are equilateral triangles they belong to the regular system, but they belong to the quadratic system if their faces are isoceles triangles. Pyramids of the hexagonal system have hexagonal bases, and their faces are isoceles triangles. Pyra-

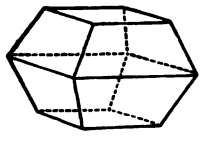


Fig. 77. Truncated octohedron. (Potassium ferrocyanide.)

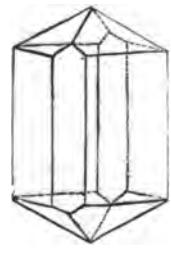


Fig. 78. Quadratic prism with pyramidal ends.

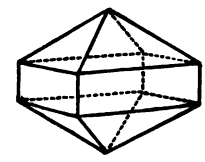


Fig. 79. Stannic oxide.

CRYSTALLINE FORMS OF THE RHOMBIC SYSTEM.

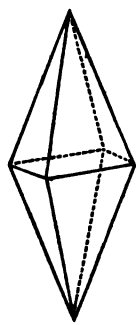


Fig. 80. Rhombic double pyramid. (Octohedron.)

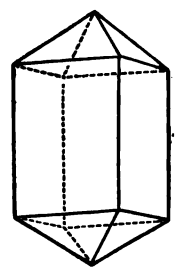


Fig. 81. Rhombic prism with pyramidal ends. (Zinc sulphate.)

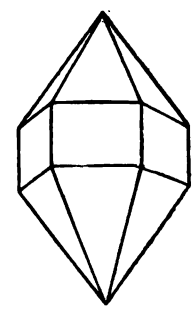


Fig. 82. Rhombic pyramidal prism. (Potassium sulphate.)

mids with rhombic or rhomboid bases belong to the rhombic system if all the axes are at right angles; to the monoclinic system if any two axes are at right angles, but not all; and to the triclinic system when there are no right axial angles.

Tetragons have four angles or corners; *hexagons* six; *octagons* eight, etc.; *tetrahedrons* have four sides; *hexahedrons* six; *octohedrons* eight; *dodekahedrons* twelve, etc.

140. Some substances crystallize with water; others without "water of crystallization." *Hydrous* crystals are those containing water; anhydrous crystals do not contain it.

Hydrous crystals may, however, be quite dry in the sense that they do not contain any moisture on their surface. On the other hand, anhydrous crystals may contain small quantities of *interstitial water* imprisoned between the individual small crystals of which larger crystals always consist. Crystals containing interstitial water *decrepitate* or burst with a slight explosion when heated.

CRYSTALLINE FORMS OF THE MONOCLINIC SYSTEM.

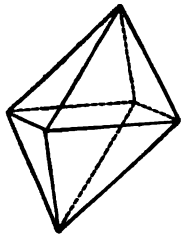


Fig. 83. Monoclinic double pyramid.

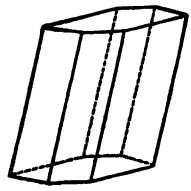


Fig. 84. Monoclinic prism of sodium acetate.

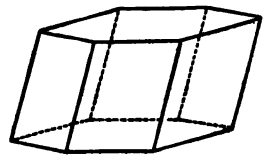


Fig. 85. Monoclinic prism of cane sugar.

CRYSTALLINE FORMS OF THE TRICLINIC SYSTEM.

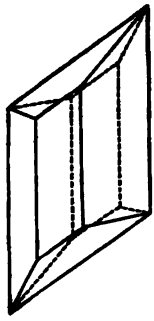


Fig. 86. Triclinic pyramid (gypsum).

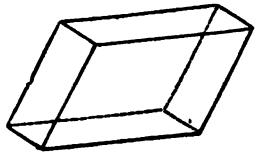


Fig. 87. Triclinic prism.

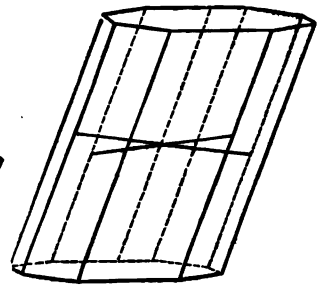


Fig. 88. Triclinic prism of calcium thiosulphate.

141. The water of crystallization is held by molecular attraction, it is essential to the crystalline form of hydrous crystals, and is always a molecular proportion.

The proportion of water of crystallization of salts varies from 5% to 60%.

Hydrous crystals are generally formed in water-solutions of the crystallizing substances, and some salts take up varying proportions of water of crystallization according to the degree of

concentration of the solutions. Crystals are, of course, formed only in saturated or supersaturated solutions, and the strength of a saturated salt-solution depends upon its temperature. Crystals formed in hot (and, therefore, stronger) solutions generally take up less water than those formed in cold solutions.

Manganous sulphate crystallized from a solution saturated at or below 6° contains seven molecules of water; crystallized from a solution saturated at from 7° to 20° it contains five molecules; at 20° to 30° it crystallizes with only four molecules of water.

Sodium phosphate crystallizes with twelve molecules of water at about 30°; but with only seven molecules from a solution saturated at 40°.

Sodium carbonate crystallizes with either ten, nine, seven, or five molecules of water of crystallization according to the temperature and strength of the solutions.

Copper sulphate generally crystallizes with five molecules of water. But if an effloresced crystal of nickel sulphate be added to a supersaturated solution of copper sulphate, crystals of the copper salt containing six molecules of water are deposited. If, on the other hand, a crystal of ferrous sulphate be added instead of nickel sulphate, the crystals of copper sulphate obtained will contain seven molecules of water.

Zinc sulphate crystallized at the ordinary room temperature, or at any temperature below 30°, contains seven molecules of water; but crystals formed at over 30° contain only five molecules.

These facts emphasize the necessity of regulating the temperature and degree of concentration of solutions from which substances are to be crystallized.

142. Salts containing water of crystallization do not always hold all of that water with the same force.

Magnesium sulphate gives up one molecule of water when dried at 30° to 52°; at water-bath heat it loses four additional molecules; and at a still higher temperature it is rendered anhydrous.

Potash alum contains about 45.6 per cent of water of crystallization; heated at 40° it gives up 2.7 per cent of that water; at 47° it loses 9.6 per cent; at 60° it loses most of its water; but long continued heating at 100° is necessary to expel all.

Crystallized ferrous sulphate, $FeH_2SO_5.6H_2O$, gives up nearly all of its six molecules of water of crystallization at from 90° to

100° ; but it begins to give off some of it even at the ordinary temperature of the air.

Sodium phosphate, containing twelve molecules of water, loses five molecules of that water at 40° to 50° ; all of it at 100°.

Efflorescence means the loss of crystalline form through the loss of the water of crystallization, the crystals falling to powder.

Hydrous crystals are frequently efflorescent, but rarely deliquescent.

Solids which neither effloresce nor deliquesce are described as "permanent in the air."

The solution of hydrous crystals in their own water of crystallization is called *aqueous fusion.*

143. The **objects** of crystallization are: 1, the separation of crystallizable substances from amorphous substances when they occur together in one solution. (This separation can also be effected by *dialysis*) ; 2, the separation of heteromorphous substances from each other when together contained in one solution; 3, the purification of commercial chemicals; 4, improvement of the appearance of the products.

144. Crystals are most readily formed when crystallizable substances pass from the liquid or the gaseous condition to the solid state, for the molecules of fluids are more mobile than those of solids, and the formation of crystals is the arrangement of the molecules into solids of definite form according to the nature of each individual crystallizing substance.

But crystallization nevertheless does take place even in solids which thus become changed from the amorphous to a crystalline condition. Some metals are known to undergo this change; also arsenous oxide, melted sugar, etc.

145. Methods of effecting crystallization. The usual means of inducing solids to assume the crystalline form are: 1, insoluble but fusible crystallizable solids are liquefied by fusion and then allowed to cool slowly (par. 29) ; 2, volatile crystallizable solids may be crystallized by sublimation; 3, soluble substances are crystallized by deposition out of their solutions; 4, some liquids are crystallized by freezing (for purposes of purification), as, e. g., glycerin, chloroform, etc. (Pictet's process) ; and 5, many insoluble, or very sparingly soluble, substances are obtained in minute crystals when produced by precipitation.

CHAPTER X.

CRYSTALLIZATION FROM SOLUTIONS.

146. The most common and successful method of crystallization is that of soluble solids by inducing them to form crystals from their solutions.

Any crystallizable soluble solid may be comparatively easily crystallized from its solution, unless the substance is so extremely freely soluble as to be nearly or quite deliquescent.

A crystallizable substance separates from its solution in the form of crystals whenever the amount of solvent present is insufficient to retain all of it in solution.

The ratio of solubility of a given solid in a given solvent at any given temperature is a constant ratio. A saturated solution of a crystallizable substance, therefore, must deposit crystals whenever the proportion of solvent is diminished by evaporation.

Crystals are also deposited when the temperature of a saturated solution is lowered if the crystallizable substance dissolved is soluble in greater proportion at a higher temperature.

Crystallization from saturated solutions is, therefore, effected either, 1, by *evaporation of the solvent;* or 2, by *reduction of the temperature of the solution;* or 3, by both of these means together.

147. Water-soluble chemical compounds are *generally* produced in a state of solution, and are recovered from their solutions by crystallization if they are crystallizable solids.

148. The **size of crystals.** Some substances naturally form large crystals, while others form small crystals. But much can be done to increase or diminish the size of crystals formed from solutions.

If *large and well developed crystals* are desired their formation must be slow. Hence they must be produced by very slow evaporation of the saturated solution, or by very slow cooling of it. As experience has further shown that large crystals are more readily obtained from weaker solutions than from stronger ones, it follows that the best plan is to make the solution saturated at not above the ordinary laboratory temperature and to expose

that solution to spontaneous evaporation. Another condition necessary to the formation of large crystals is perfect rest and abundant room for the crystals to grow in. Finally, if the temperature of the solution be as nearly constant as possible so that spontaneous evaporation of the solvent is the only means of inducing the necessary supersaturation which must precede the deposition of crystals, the most favorable conditions are attained. Sudden or great fluctuations of temperature interfere seriously with the growth of crystals.

If *small crystals* are preferred they should be caused to deposit quickly. Strong solutions, quickly cooled; rapid evaporation or vaporization of the solvent; agitation of the liquid—these means produce small crystals.

Small crystals (granulated crystalline products, turbidated salts, and products in the form of crystal meal, or in powder) are very convenient because small quantities of them can be very easily and accurately weighed and because they are quickly soluble; but some substances are so injuriously affected by contact with air that they keep very much better in large crystals than in crystalline powder.

149. *Turbidation.*—Salts which are far more soluble in hot water than in cold water may be purified or recrystallized by making saturated hot solutions of them and agitating these solutions during the process of cooling. Solutions for this purpose should be so made as to be saturated at the boiling point if they do not require filtration. But if they must be filtered, they should be made weaker in order to prevent crystallization during the process of filtration, unless facilities are available for filtering liquids at the boiling point of water. When salt solutions heated to the boiling point are to be filtered through paper filters in glass funnels it is best to make the solutions of such strength that they are saturated at about 80° C., and the funnel used should be made hot by means of hot water before the filter is inserted and the solution poured into it.

Turbidated salts are crystalline, but the crystals are small. Turbidation is the granulation of water-soluble salts by the rapid cooling of hot saturated solutions, the formation of small crystals being insured by agitation of the solution and not by evaporation.

Turbidation is applicable in the preparation of small crystals

of chlorate, nitrate, and dichromate of potassium, borax, alum, ferrous sulphate, lead acetate, copper sulphate, and many other salts.

Turbidated salts which are not affected by alcohol but which do not bear long exposure to air while in a moist state should be washed with alcohol before they are dried, as, for example, ferrous sulphate.

150. *Granulation.*—Water-soluble salts may be granulated by evaporating their water-solutions to dryness during constant stirring after the salt has begun to separate. The solution must be filtered before it is evaporated, and the evaporation is carried on without much stirring until a pellicle begins to be formed or until salt separates on the dish just above the surface of the liquid; during the subsequent evaporation the solution must be stirred to prevent the formation of larger crystals.

Salts containing water of crystallization can not be granulated by evaporation to dryness because of the danger of expelling a part of that water.

In the evaporation of salts to dryness it is necessary to take into account that some of the salts of volatile and comparatively weak acids, and the salts of ammonium, may be partly decomposed by the heat. Thus a solution of neutral ammonium sulphate might become acid during the progress of the evaporation, while a solution of potassium acetate might become alkaline. These difficulties are corrected by neutralization.

Granulated salts are generally crystalline, but many are simply granular powders without crystalline form.

Many halides, citrates, tartrates, and other salts may be granulated in this manner.

151. The bulk or quantity of product made has great influence upon the size and perfection of form of crystals, larger and finer crystals being more easily obtained the greater the quantity operated upon. Small quantities of solution sometimes refuse to give good crystals of substances which crystallize well from larger bodies of liquid.

152. When crystallization is resorted to as a means of elimination of soluble impurities, the process should be rather slow and therefore performed from solutions saturated at not above the ordinary room temperature.

153. When the object of the crystallization is simply to con-

vert an already pure product into good crystals, and the quantity of solid to be crystallized is so large as to render it desirable to divide it into two or more portions, water is used as the solvent only on the first portion, the solutions are made at a somewhat elevated temperature, the crystallization is effected by cooling, and the mother-liquor is used as the solvent for subsequent portions of the salt.

154. In manufacturing it happens most frequently that the solutions obtained when water-soluble solids are produced are rather dilute, or at all events not saturated solutions. If the product is to be crystallized it is then necessary to concentrate the solution by evaporation, or by "boiling it down."

Solutions of moderately soluble salts may be evaporated until a *pellicle* forms on the surface of the solution, or until a small sample of the solution becomes turbid from the deposition of small crystals on cooling, or until solid particles (crystals) begin to form on the sides of the vessel near the level of the liquid.

Freely soluble salts can not be well crystallized from solutions saturated at a high temperature.

155. Larger crystals are usually formed in turbid solutions than in clear ones. But clean, pure products can not be obtained from dirty solutions.

A powdered crystallizable salt when placed in a saturated solution of the same substance, gradually assumes a distinct crystalline form. The small particles of salt serve as *nuclei* for the formation of crystals which may grow to a considerable size. The size of crystals may be increased in a similar way. This is called *nursing* the crystals. Gradual changes of temperature of the solution containing an excess of (undissolved) salt promote the growth of the crystals, because the smallest particles redissolve when the temperature is increased and the dissolved salt deposits on the surface of the larger particles (or crystals) when the temperature falls.

The vessel containing the solution and salt may, therefore, be put in a warm place occasionally to effect the result described.

156. *Stunted crystals* are generally obtained when the crystallization progresses too rapidly, for the numerous crystals formed simultaneously are apt to crowd each other. The same result is, of course, also caused by contact of the crystals with the bottom and sides of the vessel.

157. The crystals formed may be either free (detached from each other), and are then usually well developed; or they may form clusters, clumps, crusts, or cakes.

Detached crystals and clusters of crystals are formed in the body of the solution where they have room and freedom to develop.

Crusts and cakes of imperfect crystallization are formed on the sides and bottom of the vessel.

To obtain particularly perfect crystals for the purpose of examining their form, a few crystals may be slowly nursed to perfection in a small vessel, each crystal being turned occasionally, or the crystal may be suspended in the solution by a thread so as to be free to develop in every direction.

When large quantities of solution are made to deposit crystals a mass of fine, large, free crystals are usually formed in the center, while crusts are formed on the sides and bottom of the vessel.

158. Retarded crystallization. One of the inconveniences attendant upon the crystallization of salts from solutions is the formation of supersaturated solutions which sometimes refuse to deposit crystals. This can generally, but not always, be remedied by dropping some crystals of the same salt into the supersaturated solution. When once started the crystallization usually continues without interruption.

The addition of another salt is a remedy rarely practicable.

But retarded or difficult crystallization may sometimes be advantageously prevented by the very gradual addition of a non-solvent liquid to the solution. Thus, if some alcohol is very cautiously laid over the surface of a strong water-solution of a substance nearly insoluble or only sparingly soluble in alcohol, the water will gradually absorb the alcohol and give up the salt which then crystallizes if the process is slow enough.

Substances crystallizing with a large amount of water of crystallization sometimes fail to form crystals when the solutions are too strong, or when the required amount of water is not present, as is the case with ferric chloride.

159. Crystallizers. The vessels in which crystallization is effected are called crystallizers. They may be deep if the crystallization is to be induced by lowering the temperature of the

saturated solution. But if the crystallization is to be effected by spontaneous evaporation the crystallizer should be shallow.

Experience has shown that when large quantities are operated upon, angular crystallizers and those with a comparatively rough interior surface are preferable to smooth, spherical vessels. Glass and glazed porcelain are, therefore, not always the best materials out of which crystallizers are made. Circular or rectangular flat-bottomed dishes of glass and of porcelain are very good for small operations, but large crystallizers are made of wood, rough earthenware, iron, and other material according to the nature of the substances to be crystallized in them.



Fig. 89. Crystallizer of glass.

160. Points of attachment for the crystals are useful if the crystallization requires to be hastened by such means, as when the crystals are to be made of large size and the process is accordingly otherwise slow.

Just as crystallizers with angles or corners, or with a rough interior, are advantageous for facilitating crystallization, so are sticks, strings, or wires placed in the liquid an excellent means to the same end, whenever their introduction is admissible. Rock candy, ferrous sulphate, copper sulphate, and potassium ferrocyanide are crystallized on a large scale in that way.

161. Mother liquors. The saturated solution remaining at the end of the deposition of crystals, and, in fact, the liquid in which any crystals are formed, is called the *mother liquor.*

When the solution contains a perfectly pure salt, the only object being the recovery of that salt or its conversion into crystals, the mother-liquor is, of course, generally still a solution of pure salt at the end of the process. But if the mother-liquor is evaporated from time to time with the aid of heat, the last mother-liquor may not be free from impurities, for the heat may cause more or less decomposition, as it usually does in the concentration of consecutive mother-liquors obtained in the crystallization of organic substances (for instance, citrates, tartrates, alkaloidal salts, etc.).

162. Whenever the crystallization is effected by the cooling of

solutions made saturated at an elevated temperature, and the mother-liquor after each crystallization is concentrated by evaporation with the aid of heat, several **crops of crystals** are obtained—one from each operation. In all such cases the probable result will be that the first crop is better than any subsequent one, and that the last crop is poorer than the preceding ones.

163. Whenever crystallization is performed for the purpose of purifying the substance to be crystallized, each successive mother-liquor must contain an increasing proportion of the impurities, and each successive crop of crystals must be more and more liable to be impure, until finally the *end-mother-liquor* is reached from which a product fit for use without further purification can not be obtained.

Usually only three crops of satisfactory product can be obtained.

But the end-mother-liquor, if of sufficient value, can be purified by various means according to its nature, and its contents recovered or utilized in one way or another. For instance, when the mother-liquor from Rochelle salt has been concentrated several times it finally becomes dark-colored from finely divided carbon, and a white or colorless salt can then no longer be obtained from it; but if the end-mother-liquor be rendered strongly acid by the addition of hydrochloric acid to it, the valuable tartrate radical is recovered in the form of cream of tartar.

164. Creeping salts. In the evaporation of solutions of certain ammonium salts, and a few salts of potassium and sodium (as, for instance, the benzoates) for the purpose of granulating or crystallizing these salts, it happens that the salt is deposited on the sides of the evaporating-dish above the level of the liquid, and that the solution creeps up by capillary action between the particles of salt and the crust thus formed by evaporation of this solution extends over the top of the dish and on its outside unless the salt is scraped down from time to time. But the "creeping" may be prevented by slightly greasing the dish.

165. The crystals may be **collected** with casseroles, scoops, ladles, spoons, or otherwise, according to their quantity. Small crops are usually loosened from the crystallizer and poured into a funnel with the mother-liquor. When very small quantities are crystallized the mother-liquor may be allowed to evaporate to dryness.

166. The wet crystals must be **washed** if the mother-liquor is dirty or impure. For this purpose they are placed in a perforated funnel or draining cone, and, *after the mother-liquor has run off,* the mass of crystals may be hastily rinsed with a small amount of cold distilled water, or with a little alcohol if not objectionable on chemical grounds. Crystals washed with alcohol are easily dried.

Before washing the crystals with water, it is necessary to break up any clumps.

The water used in washing the crystals usually spoils their appearance somewhat by taking off the sharp edges. To avoid this they may be washed with a pure saturated solution of the same substance.

Crystals formed in solutions made from pure substances, of course do not need washing.

167. Wet crystals and crystalline products are **drained** in perforated funnels, or draining cones, or on muslin strainers. When small quantities of crystals are formed in dishes or beakers these vessels may be tilted in such a way as to allow the mother-liquor or wash-water to run off. The wet crystals or mass should be allowed to stand long enough to become thoroughly freed from liquid, so that they are nearly or quite dry, unless greater despatch is necessary.

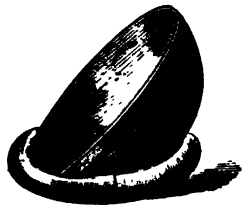


Fig. 90. Position of dish for draining of crystals.

The Buchner funnel (fig. 18) and the centrifugator (fig. 91) are also used in freeing crystals from water.

Small quantities of wet or moist crystals may be drained or even dried on porous tiles or on pure white blotting paper or filter paper. But handsome, slender crystals should not be pressed between paper or cloth if they are so frail as to be broken or crushed by that treatment.

168. The **drying of crystals** is a very important matter.

Substances which are permanent in the air and not affected by moderate heat may be very easily dried in layers about 15 to 20 millimeters thick on plates of glass. Double thick window glass is suitable.

The product should be frequently stirred or turned with a porcelain spatula or other suitable stirrer.

Hydrous crystals should be dried at temperatures not above 30° to 40°.

Efflorescent and deliquescent hydrous salts are liable to be partially dissolved in their own water if dried at a too high temperature, and this would greatly damage if not ruin the product.

Alcohol-washed crystals dry readily.

Anhydrous crystals may in most cases be dried at 100° to 120°; but if this is not convenient they may be exposed a longer time and dried at a lower temperature.

When crystalline or other salts are dried with the aid of heat, it is necessary to know whether or not they are in any way changed or injured by the heat to which they are exposed, and the temperature must be regulated accordingly.

Sodium bicarbonate when dried with the aid of heat is liable to give off CO_2 as well as water. Acetates dried with or without the aid of heat are liable to lose acetic acid. Lead acetate in very small crystals should be dried in an atmosphere of acetic acid vapor.



Fig. 91. A centrifugator for removing mother liquor and water from crystals and precipitates.

Crystals dried by exposure to the air are liable to be of dull appearance.

Crystals may be quickly dried by centrifugation. When draining is impracticable, as when the adhering mother-liquor is dense or syrupy, the drying is effected in rapidly revolving perforated drums. Granulated sugar is dried in that way. The small centrifugal machine figured in this text may sometimes be used to advantage for drying efflorescent salts and other products difficult to dry in the usual simple way (by exposure to the air).

Desiccators are rarely used for drying inorganic substances. They are glass bells placed upon plates of glass over vessels containing fused calcium chloride, calcium oxide, concentrated sulphuric acid, or other very hygroscopic substances which absorb the water vapor given off from the crystals or other substances placed above, or beside them. See fig. 53.

169. Before being **bottled** the product must be perfectly dry,

if possible, and the bottle in which it is to be put must be perfectly dry and warm. After the bottle is filled it should at once be tightly closed.

170. Physical precipitation. The separation of solids from their solutions may be effected by the addition of liquids *miscible with the solvent* but in which the substances contained in the solutions are not soluble.

Many water-soluble salts which are insoluble in alcohol may thus be precipitated or separated in the form of small crystals by the addition of sufficient alcohol to their respective water-solutions.

Sometimes this method of obtaining salts in a granular condition or in minute crystals is also at the same time an effective method of purification when the impurities are soluble in the precipitant, as in the preparation of precipitated ferrous sulphate.

Substances soluble in alcohol but insoluble in water may be precipitated from their alcoholic solutions by means of water.

Physical precipitation usually produces a crystalline precipitate when the product is an inorganic salt. When ferrous sulphate, copper sulphate, tartar emetic, and certain other water-soluble salts insoluble in alcohol are precipitated by adding alcohol to their strong water-solutions, or by pouring the solutions into alcohol, the crystalline precipitates thus obtained are easily and quickly dried because wet with alcohol instead of water.

CHAPTER XI.

DIALYSIS.

171. Dialysis is a process by which "crystalloids" may be completely eliminated from "colloids" when both kinds of substances occur together in water-solutions. It is the diffusion of crystalloid matter in aqueous solution through a suitable organic septum. The septum generally used is parchment paper. This is tied tightly over a circular frame of glass, porcelain, hard rubber, or wood, so that the frame forms a vessel resembling a sieve of which the parchment paper is the sieve cloth or bottom. This apparatus is called a *dialyser,* and when it is used the solution which is to be subjected to dialysis is placed in the dialyser which is suspended in a large vessel containing water, as shown in the cut.



Fig. 92. Dialyser.

172. Crystalloids are substances which resemble crystallizable water-soluble compounds in that they pass through such septa as parchment paper when in a state of solution in water and placed in a dialyser.

All water-soluble crystallizable salts are crystalloids; but crystalloids are not necessarily crystallizable.

Colloids (from *collum,* glue, and *eidos,* like) are water-soluble substances which, like glue, gelatin, gum, etc., are unable to pass through the dialyser in the manner described.

Crystalloids and colloids can, therefore, be separated from each other by dialysis.

173. Dialysis is employed in chemical processes to separate inorganic crystalloids from organic colloids, for the purification of certain chemical products, etc.

Arsenical compounds, lead salts, poisonous alkaloidal salts, etc., taken into the stomach may be completely dialysed out of the mixture of the total contents of that stomach, and then easily separated and identified.

Salicylic acid may be freed from impurities by dialysis.

The preparation called "dialysed iron" is a water-solution of a compound formed by saturating a dilute solution of ferric chloride with ferric hydroxide and then removing by dialysis whatever ferric chloride remains unchanged, together with the ammonium salt left in the ferric hydroxide should this be incompletely washed (or not washed at all).

The liquid passing out of the dialyser is called the diffusate.

To perform dialysis successfully the dialyser should not be filled, but should contain a quantity of liquid not over 10 to 15 millimeters in depth.

CHAPTER XII.

PRECIPITATION.

174. Precipitation is the formation of insoluble solid matter in a liquid.

The insoluble solid formed is called a **precipitate.**

The precipitate may be absolutely insoluble in the liquid in which it is formed, or it may be so sparingly soluble as to be "practically insoluble." It may be nearly insoluble in that liquid at the ordinary room temperature, but more soluble in the same liquid at a higher temperature, and may be very soluble in other liquids.

Precipitation occurs only in liquids.

175. It is important that distinction be made between *physical precipitation* and *chemical precipitation.*

Physical precipitation occurs when a sufficiently strong solution of a solid substance is mixed with a sufficient proportion of some liquid miscible with the solvent but in which the dissolved substance is insoluble. The dissolved substance is then more or less completely thrown out of its solution, re-assuming the solid state. No chemical reaction takes place in physical precipitation, for all the substances contained in the liquid before and after the precipitation, including the solid substance precipitated, were already present or added and no new substance is formed.

Chemical precipitation results from the formation of new molecules in a liquid, or a mixture of liquids, one or more of the new substances being insoluble in the liquid or mixture. Chemical precipitation is, therefore, always caused by chemical reaction, and the precipitate is in all cases of chemical precipitation an insoluble product of that reaction. The chemical reactions by which precipitates are formed may or may not be complete according to the proportions of the reacting factors and according to other circumstances attending the precipitation.

It will be seen from the foregoing that physical precipitation results from a change in the solvent by which it is converted into a non-solvent, while the substance originally held in solution, and,

therefore, in a liquid condition, is simply separated in a solid state; chemical precipitation, on the contrary, is caused not by any change in the liquid which constitutes the solvent of the original solution, but by a chemical change in the substances dissolved.

176. The separation of solid matter from a solution previously free from solid particles is not precipitation if the solid matter is soluble in the liquid and is separated for want of a sufficient proportion of the solvent to retain it in solution.

The crystallization of a salt from its solution differs from precipitation in several ways. Crystallization takes place more slowly, whereas precipitates are generally formed precipitately. Crystals are formed in saturated solutions by a reduction of the temperature or by loss of a portion of the solvent by evaporation, and they grow by further cooling or evaporation of the solution in which they were formed; but precipitates are formed in dilute as well as in strong solutions, generally without reference to the temperature of the liquids in which they are formed, and the particles of solid matter constituting the precipitate do not increase in size unless crystalline and contained in a saturated solution of the same substance.

Amorphous solids may, also, separate from saturated solutions by lowering the temperature or by the evaporation of the solvent, and depositions of amorphous solids from their solutions are always called precipitation, while the formation of crystals in liquids may not properly be called precipitation unless the crystals are formed as the result of chemical reactions and formed precipitately. The slow formation of precipitates is exceptional.

177. Chemical precipitation is the chief subject of this chapter, and its great importance may be recognized from the fact that nearly all insoluble inorganic solids and many soluble ones are produced by metathesis resulting in the formation of one soluble and one insoluble product.

178. Precipitation results from the law, that, whenever, in case of metathesis between two soluble salts which are mixed with each other in a state of solution, an insoluble product (or a product less soluble than either of the factors) would be formed, then that metathesis will certainly take place and will proceed to completion.

179. The materials required for the production of insoluble

chemical compounds by precipitation are, with rare exceptions, *soluble compounds.* Hence the compounds of potassium, sodium (and ammonium) are very largely employed to furnish the negative radicals of the insoluble inorganic products, and the sulphates, nitrates and chlorides of the other metals are the most common materials furnishing the positive radicals (the metals) toward the formation of the same products. (See Chapter XIX, Vol. I.) Thus the factors of the reactions are chiefly salts. They must, of course, be of satisfactory quality.

180. Proportions of the factors. While the relative quantities to be taken of the materials are always computed on the basis of the number of molecules required of each to complete the reaction (as shown by the chemical equation representing it), it must be borne in mind that the exact proportions thus found, although absolutely correct in theory, rarely give satisfactory results, because in the production of insoluble compounds by precipitation it is almost invariably necessary that the factor supplying the positive radical of the insoluble product shall be completely decomposed, and for this reason the other factor of the reaction must be used in excess of the proportion indicated by theory.

Thus, when an insoluble iron compound is to be prepared by precipitation the iron salt used for that purpose must be completely decomposed; if a mercury compound is to be made all of the mercuric chloride used must be consumed; if a lead compound is prepared out of lead nitrate not a trace of the lead nitrate must be left over. Experience has shown that if an insoluble compound of any given metal, when it is being formed by precipitation, is allowed to come in contact with a soluble compound of the same metal contained in the liquid in which the reaction is effected, the composition of the product sought may not be normal. Pure mercuric iodide is never formed when solutions containing the exact theoretical proportions of mercuric chloride and potassium iodide are mixed; pure mercuric oxide is not formed when the exact theoretical proportions of mercuric chloride and sodium hydroxide are mixed; pure normal ferric hydroxide is not obtained when ammonia is added to a solution of ferric sulphate.

An *excess* must, therefore, be used of that factor which sup-

plies the negative radical of the precipitate in order that every molecule of the other factor may be decomposed.

To insure that a pure lead iodide is obtained from lead acetate and potassium iodide, an excess of the potassium iodide is necessary in order that no lead acetate may be left undecomposed, for if lead iodide in the act of its formation as a precipitate comes in contact with lead acetate in solution the precipitate will not consist of pure lead iodide but of so-called "oxy-iodide" of lead.

How much of an excess is actually necessary it is difficult to state. Perhaps from three to five per cent is a sufficient addition to the quantity theoretically required; but in cases where experience has shown that the tendency toward the formation of meta-compounds, "basic compounds," double-salts, and other abnormal or undesired products, is great, an excess of even ten per cent above the quantity called for according to the chemical equation may be requisite. A considerable excess is safer than a very small one, and no disadvantage can attend the use of a larger quantity than is really necessary except that it would be an unnecessary waste. The practical question before the operator is simply which of the two evils he will choose—a slightly increased cost of production without any risk of spoiling the product, or the reduction of the cost of production to a minimum coupled with the danger of obtaining an unsatisfactory product. In this connection it is well to take into consideration the fact that in the production of inorganic chemicals the necessary labor and skill are quite generally worth much more than the cost of the materials.

181. The solutions used for making precipitates must be perfectly clear. *Filtration* is almost invariably necessary to insure this.

It may be said that, within certain limits, dilute solutions are more favorable to free and complete chemical interaction than strong solutions. But the *strength,* or the degree of concentration or dilution, which ought to be fixed upon in each case must be determined by several circumstances. In some cases the materials employed may not be sufficiently freely soluble to admit of the use of any other than weak solutions.

Comparatively dilute solutions should always be made of any salts the complete decomposition of which is to be insured; and stronger solutions should be made of any alkalies or salts which

are to be present *in excess* in the liquid in which the reaction takes place. Thus when ferric hydroxide is made from solutions of ferric chloride and ammonium hydroxide, the iron solution should be weak but the ammonia less dilute. When yellow oxide of mercury is made the solution of sodium hydroxide need not be very diluted, and, in fact, *ought not* to be so, but the solution of mercuric chloride *must* be weak.

Dilute solutions, as a rule, make light or bulky, finely divided precipitates, while strong solutions produce heavier or denser, coarser, precipitates.

But *experience* is the best guide. Therefore, unless the laborant has had sufficient experience of his own, he should be guided by that of others. The directions given in reliable laboratory manuals are based upon the experience of many operators.

The *temperature* of the solutions is also a matter of considerable importance in many precipitations. Some precipitates are liable to be decomposed or otherwise injuriously affected by heat and, therefore, require to be made from cold solutions. Cold solutions frequently produce more bulky and finely divided precipitates where hot solutions make denser products.

182. The order of mixing the solutions is often one of the necessary conditions of success.

It is necessary to make proper distinction between the two solutions employed for the production of precipitates by metathesis, and the only technical terms I find in use are the words "precipitant" and "precipitand." The solutions are necessarily mixed by adding one to the other. [The suggestion that both solutions be simultaneously poured together into the precipitation vessel is probably not of any value in a single instance.] The solution first put into the precipitation vessel was called the "precipitand;" and the other solution, poured into the first one, was called the "precipitant." Many have discarded or refused to adopt the term precipitand because it is scarcely possible to pronounce the two words so distinctly that one is not mistaken for the other. I shall, therefore, use the expression *primal solution* in this book, meaning thereby the solution which is put in the precipitation vessel *first;* and will retain the term *precipitant* to designate the solution which is to be added to the primal solution.

But—which of the two solutions shall we pour into the jar first? Let us consider an example. The so-called "ammoniated

mercury" of the pharmacopœias is an insoluble compound made by mixing a solution of mercuric chloride and a solution of ammonium hydroxide (ammonia water). Now, if we put the solution of mercuric chloride into the jar first and then add the ammonia water, the solution of chloride of mercury will be our primal solution and the ammonia water will be our precipitant; but if we put the ammonia into the jar first and then pour the mercury solution into the ammonia, our primal solution will be the solution of ammonium hydroxide and the solution of mercuric chloride becomes the precipitant. Will it make any difference what course we pursue? Decidedly. If we use the solution of mercuric chloride as the primal solution, and ammonia water as the precipitant, the precipitate produced will be a compound said to consist of mercuric chloramide and mercuric chloride, and usually represented by the formula $NH_2(HgCl)_2Cl$; but if we use the ammonia as our primal solution and the solution of mercuric chloride as the precipitant, then the precipitate produced will be pure mercuric chloramide, $Hg\begin{smallmatrix}Cl\\NH_2\end{smallmatrix}$

[The $NH_2(HgCl)_2Cl$ is said to be $ClHg{-}\overset{\displaystyle Cl}{\underset{\displaystyle H\ H}{N}}{-}HgCl$ in which the nitrogen is a pentad with a polarity-value of —3, the only positive nitrogen bond being that which holds the one chlorine atom in direct combination.]

If the student will now refer again to paragraph 180 he will understand that the primal solution must be the solution of that factor which contributes the negative radical toward the formation of the precipitate, and that the other factor—the one contributing the positive radical of the precipitate—must be made the precipitant.

As the scope of this book is limited to the inorganic chemical compounds, and as nearly all the insoluble inorganic compounds made by precipitation are metallic compounds, we can simplify the rule just given as follows: *An insoluble metallic compound*

should not be precipitated in a primal solution containing a compound of the same metal that enters into the composition of the precipitate.

A precipitated mecury compound should not be produced in a solution of a mercury salt; do not make an insoluble iron compound in a liquid containing a soluble iron salt; do not let an insoluble lead compound be formed in a solution or mixture containing a lead salt; a precipitated copper compound should not, in the act of its formation, be allowed to come in contact with a copper solution; do not let an insoluble zinc compound lie in a solution of a zinc salt, and do not make it in such a solution.

If ammonia water be added to a solution of ferric sulphate the reaction will be as follows:

$$Fe_2(SO_4)_3+6H_4NOH=2OFeOH+3(H_4N)_2SO_4+2H_2O.$$

But if a solution of ferric sulphate be added to ammonia water the result is:

$$Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4.$$

It is clear that if ammonia water is gradually added to a solution of ferric sulphate, the ferric sulphate will, at least at the beginning, be present in excess; but if the order of mixing the liquids be reversed, the ammonia water will be in excess. With the ammonia present in excess it is impossible to obtain any other products than $Fe(OH)_3$ and $(H_4N)_2SO_4$, for the $Fe(OH)_3$ is insoluble in and unaffected by the ammonia water and the ammonium sulphate; all of the ferric sulphate is decomposed as fast as it is added, and there is accordingly no ferric salt present in the solution at any time. But when the solution of ferric sulphate is used as the primal solution and the ammonia water as the precipitant the ferric hydroxide, $Fe(OH)_3$, first formed redissolves in the solution of ferric sulphate:

$$2Fe(OH)_3+5Fe_2(SO_4)_3=3Fe_4O(SO_4)_5+3H_2O.$$

Subsequently, when more ammonia is added, the "basic ferric sulphate" is decomposed:

$$Fe_4O(SO_4)_5+10H_4NOH=$$

$$4OFeOH+5(H_4N)_2SO_4+3H_2O.$$

Assuming that the proportions of the factors are correct, we know that, when one liquid is added to the other, the precipitant is completely decomposed as fast as added, and the primal solution remains in excess from beginning to end, provided other conditions are favorable.

183. To insure complete decomposition of the precipitant as fast as it is added to the primal solution, it is necessary to observe the following precautions:

a. The precipitant should be a rather dilute solution, while the primal solution should be less diluted (but not concentrated).

b. The precipitant should be added slowly, or gradually, and this is best accomplished (when great caution is necessary) by running it into the primal solution through a syphon of small diameter. Or the precipitant may be added in small portions, one portion at a time.

c. The primal solution should be well stirred, as the precipitant is added, and brisk stirring of the mixture should be continued without interruption until all of the precipitant has been used.

It may be readily seen that if the precipitant be added too rapidly, in rather large quantities, and without stirring, some of the newly formed precipitate may come in contact with portions of the undecomposed precipitant, which is the danger to be particularly guarded against.

184. When very concentrated solutions are used the reaction is liable to be incomplete, especially if the precipitate is bulky.

The precipitation is generally more complete when hot liquids are used; but hot solutions should not be employed unless it is certain that the precipitate will not be injuriously altered by the higher temperature.

185. Occasionally the proportions of the factors of the reaction can not be fixed beforehand in accordance with their molecular weights. This happens when the quantity of the compound contained in the primal solution is unknown. The precipitant is then to be added cautiously, a small portion at a time, until it ceases to cause further precipitation. To guard against adding an excess of the precipitant in such cases it is best to test

the liquid from time to time by filtering off a test sample and testing that with a drop or more of the precipitant. Complete and even precipitation may thus be accomplished *if admissible;* but it is generally best to stop the further addition of precipitant before quite all of the other factor of the reaction (that contained in the primal solution) has been decomposed, and in no case should an excess of the precipitant be used unless requisite for some special reason or known to be proper.

186. The precipitate. Precipitates vary greatly in their physical properties. They may be crystalline or amorphous, heavy or light, dense or bulky, coarse or fine; they may be granular, or curdy, or flocculent, or gelatinous. A very bulky, finely divided, amorphous, almost pasty or gelatinous precipitate tenaciously retaining much water, is called a *magma.*

We have said that hot and strong solutions are likely to produce denser, heavier and coarser precipitates, whereas cold and dilute solutions tend to produce bulkier, lighter and finer precipitates. But the denser precipitates formed in hot liquids generally become bulkier if allowed to remain in the liquids long after they have become cold, and bulky precipitates become denser if left a long time in the liquids in which they are formed. Precipitates should, therefore, be collected and washed as expeditiously as practicable if it is desired to prevent such changes. The hydroxides, carbonates and phosphates of calcium, magnesium, and iron, and aluminum hydroxide, when precipitated in the cold, are so bulky and finely divided as to be nearly gelatinous, and they are, therefore, precipitated from hot solutions, instead; but if they are allowed to remain in the liquid until it becomes cold, and washed with cold water, the object of the use of hot solutions is at least partially defeated.

Precipitates which contain water, or from which water may be split off by rearrangements of the interatomic linking, vary in composition according to the temperature of the liquids in which they are formed. This variableness may be observed in magnesium carbonate, the carbonates of zinc and of lead, ferric hydroxide, and several other compounds. Precipitates made and washed in hot liquids sustain a loss of water; those made and washed in cold liquids do not. On the other hand, some precipitates take up water when long exposed to its action at the ordinary room temperature, or they may undergo other changes.

Bismuth subnitrate, when allowed to remain wet too long, or when it lies in a large quantity of water, becomes denser, coarser and "more basic" (that is, its composition changes so that the proportion of metal in it seems to be increased, probably by the splitting off of water and acid).

Whenever the precipitate is to any extent soluble in the liquid in which it is formed and is at the same time capable of assuming a crystalline form, the use of hot liquids may cause the product to be coarser and more distinctly crystalline. This is sometimes desirable as a means of improving the appearance of the product. In such a case it is necessary to let the liquid cool gradually, but thoroughly, to render the crystals larger, and to reduce to a minimum the amount lost by being retained in the liquid.

187. The inorganic compounds most frequently made by precipitation are the oxides, hydroxides, sulphides, carbonates, oxalates and phosphates of the heavy metals, the carbonates and phosphates of calcium, strontium, barium and magnesium, the halides of silver, lead iodide, mercurous chloride and iodide, and mercuric iodide.

188. Precipitation vessels. The best precipitation vessels for



Fig. 93. Precipitation jar.



Fig. 94. Tubulated decantation vessel.

large operations are stoneware pots, which may conveniently be of from 5 liters to 250 liters capacity. When larger vessels are required wooden tanks are often suitable. Tubs and barrels are also employed.

Special so-called "precipitating jars" of glass, porcelain, or other earthenware, are sometimes used. They are tall, round vessels, wider at the bottom than at the top, as shown in the cut.

Some precipitation and decantation vessels are tubulated, or provided with spigots or taps at various heights, as shown in fig. 94 to serve as outlets for the removal of the supernatant liquid whenever the precipitate has subsided sufficiently.



Fig. 95. Beaker with lip.



Fig. 96. Beaker without lip.



Fig. 97. Precipitation flasks.

But any well glazed, acid-proof jars, or porcelain or glass vessels may be used, and for quite small quantities the ordinary Erlenmeyer flasks, beakers, and wide-mouth bottles.

189. The liquid in which the precipitate is formed, obtained by mixing the two solutions (the precipitant and the primal solution) contains the bye-product dissolved in it. It is called the *mother-liquor,* the same technical term being used to designate this liquid as is given to the liquid from which the crystals are deposited in the process of crystallization. If the precipitate is heavy enough to sink down to the bottom of the precipitation vessel, or at least to descend below the surface of the mother-liquor, then the clear or nearly clear liquid standing above the precipitate is called *the supernatant liquid.*

It is in some cases important to remove the precipitate from the mother-liquor or supernatant liquid as quickly as practicable, lest the product be unfavorably affected by too long contact with it.

190. Removal of the mother-liquor or supernatant liquid is necessary before the precipitate can be effectively washed.

Should the bye-product contained in that liquid be of sufficient value to warrant its recovery it is, of course, desirable to collect the mother-liquor with as little loss as possible. After filtration the solution may then be concentrated by evaporation and the bye-product crystallized out. But owing to the necessity of employing an excess of one of the factors of the reaction the bye-product is rarely pure, and must be purified before it can be utilized. Should this be the case the mother-liquor may be boiled down to concentrate it before filtration, and the filtrate, or the residue obtained upon its evaporation, purified by whatever method may be applicable in each case. Salts recovered from mother-liquors after precipitations may often be purified by repeated recrystallizations. When the bye-product is to be recovered it is advantageous, if in other respects unobjectionable, to employ strong solutions in performing the precipitation, for the mother-liquor will then be less diluted and less concentration by evaporation will be required.

If the bye-product is not to be recovered it is nevertheless desirable to separate the mother-liquor from the precipitate as far as possible before proceeding with the washing-process, because the object of washing the precipitate is the complete removal of all of the soluble matter and this is more speedily effected by first getting rid of as much as possible of the mother-liquor than by removing only a portion of it and diluting the remainder.

Supernatant liquids, whether mother-liquor or washings, are removed from the precipitate by *decantation,* either by tilting the precipitation vessel or with the aid of the tubulures or spigots if a tubulated vessel is used, or with a syphon. The decantation of liquids and the utility of the guiding rod and greased rim have been sufficiently discussed in Chapter VI. The preparation, rigging and use of *cloth strainers,* and the use of *presses* for the separation of liquids from bulky precipitates are also described in the same chapter.

191. The washing. After the mother-liquor has been separated from the precipitate as far as practicable, the soluble matter of which a considerable quantity still remains adhering to and contaminating the product must be washed out. As the precipitation itself is generally performed with aqueous solutions and the bye-product is water-soluble, the washing is effected with water.

The water used for this purpose may be either hot or cold according to the requirements of each case. When *hot water* must or can be used, it is more effective in removing the soluble matter, so that a smaller quantity of hot water will do as much work as a larger quantity of cold water and do it more quickly. Another advantage gained when hot water can be safely used is that it renders the subsidence of the precipitate easier by making the liquid lighter, and (in some cases) the precipitate more dense.

192. *Rapid washing* is very desirable because prolonged contact with water frequently causes the precipitate to contract when this is not desired, or it may have the opposite effect when the precipitate was produced from hot liquids.

193. *Heavy precipitates* are the easiest to wash. They subside rapidly and compactly in the precipitation vessel so that the mother-liquor can be almost completely removed. The wash water is then added and the precipitate thoroughly mixed with it by vigorous shaking or stirring; the first "washings" must then be thoroughly removed before a second portion of water is added, the shaking or stirring repeated, and the second washings also separated before a third portion of water is added. By repeating the "affusion and decantation" of water several times the washing will be readily finished.

194. *Light precipitates and magmas* are not so easily washed because they do not settle down to the bottom of the precipitation vessel either rapidly or compactly. Should the precipitate remain suspended in the mother-liquor on account of the too great density of that liquor the difficulty may sometimes be remedied by dilution (with hot water if admissible), or by warming the mixture. If this method should be inadequate or inadmissible, the thick mixture may be turned into a muslin strainer and allowed to drain. The muslin strainer should first be *dipped in hot water* and wrung out before it is placed on the frame and bagged to receive the precipitate or magma. Should the liquid which passes be turbid it must be returned until it runs clear. When all of the liquid has passed through the straining cloth, the pasty magma on the cloth is returned to the cleaned precipitation vessel (or to any other suitable pot) and there thoroughly mixed with water, after which the mixture is turned into the muslin strainer again and the washings allowed to pass through. These operations are

then repeated as many times as may be necessary to complete the washing.

195. When small quantities are treated it may be best to perform the washing on a paper filter. A plain filter (one with as few folds as possible) must be used for this purpose. A folded paper filter can be made to fit a funnel of any angle by making the second fold either larger or smaller than a right angle to the first fold of the paper, according to whether the folded filter is too wide or too narrow at the top when placed in the funnel. It is best to make the angle of the paper filter slightly more obtuse than that of the funnel. Wetting the filter promotes clear and rapid filtration, because it causes the paper pulp to swell so that the pores of the paper close sufficiently not to let solid particles pass through, whereas if a fine precipitate is allowed to get into the pores before the paper is wetted the fibres may afterwards swell so as to close the pores altogether or to such an extent that no liquid will pass. The water should be added in portions and each portion allowed to pass through the precipitate and out of the filter before another portion of water is added.

196. In washing precipitates on paper filters the "spritz bottle" is useful for the purpose of directing a stream of water on the filter at the top and all around so as to wash down the precipitate into the throat of the funnel where it can be more easily covered with each portion of wash-water added.



Fig. 98. Spritz-bottle.

197. The washing may be known to have been completed when the wash-water no longer removes any more soluble matter. The presence of any considerable quantity of the soluble bye-product in the washings usually makes itself known by the taste, which is in most cases saltish. The washing-process is continued only *until the washings are tasteless* whenever circumstances render this sufficient. But when the precipitate is to be a pure finished

product it is necessary to use chemical *reagents to determine the completion of the washing* from the entire absence of the soluble substances in the end washings. The soluble substance contained in the mother-liquor and washings is known from the materials employed and the reaction which occurs between them, and the reagent required is, therefore, also known. As the materials most generally used are sulphates and chlorides the reagents for the identification of these compounds are much in demand for testing washings in the preparation of precipitated products. Thus the test solutions of barium nitrate or chloride, and silver nitrate, are the most common reagents.

198. After completing the washing-process the next step is usually that of letting the precipitate *drain* preparatory to drying it. The draining process may be effected on a cloth strainer or on a paper filter, according to the quantity. The wet precipitate is allowed to lie undisturbed on the cloth or filter until no more liquid passes; the dropping sometimes ceases while the precipitate still retains much water and in such cases it is worth while to give the strainer frame or funnel a gentle tap, which often has the effect of causing some more water to run off.

The centrifugal machine may sometimes be advantageously employed to throw off moisture from wet precipitates as well as from wet crystals.

199. The removal of drained or dried precipitates from straining cloths and filters must be effected with sufficient care. If the precipitate has been thoroughly drained it generally comes off very easily, and if allowed to dry completely on the cloth or paper it often comes off still more readily; but large bodies or thick layers of magma dry too slowly and are, therefore, generally spread out on glass plates or on tiles or stoneware dishes to dry.

When precipitates have been drained on a cloth strainer as far as practicable they may be loosened or if necessary scraped off and transferred to the plates on which they are to be dried, or to dry cloths placed on frames.

To remove precipitates from paper filters the paper must be carefully unfolded so that it may not be torn, and if the precipitate adheres to the paper, rendering it necessary to scrape it off, this must be done cautiously in order to avoid getting fragments or fibers of the paper mixed with the product. In unfolding the paper filter to remove the precipitate it is well to first place it upon

a clean cloth or sheet of paper, or in a dish, or on a glass plate.

200. The **drying** of precipitates is occasionally difficult. All products of this kind must be thoroughly dried. The drying may frequently be effected on hot plates, or on frames or plates in a drying room or drying closet. But some precipitates either do not withstand exposure to heat or dry into hard, tough cakes that can not be easily reduced to soft, fine powder. Many precipitates form soft, friable, light masses when dried at the ordinary temterature, and this is a great advantage because the cakes or lumps so obtained can easily be gently rubbed through a fine sieve cloth to form soft, fine, bulky products.

Small amounts of moist or wet precipitates which can not be dried in the ordinary way without difficulty or much delay may be spread out in thin layers on clean, new, porous, unglazed stoneware dishes, tiles or bricks, which may in many instances be used warm or even hot. Common flower pot dishes are often used to advantage. But porous dishes, tiles and bricks can, of course, not be used more than once.

Precipitates which are known to be sensitive and liable to be injured by exposure to air should be dried as quickly as practicable, and those that are injured by light must be dried in a dark place. All of them must be well protected against dust and dirt; they may be loosely covered with thin muslin or white filter paper.

201. Pulverization of precipitates. Some precipitates are micro-crystalline or granular and form coarse or moderately fine powders when dry without requiring any pulverization. But precipitates which dry in hard cakes or lumps must be reduced to very fine powder by sifting, or by trituration or grinding followed by sifting. The finer and softer the powder is the better, unless it be of perfectly uniform micro-crystalline structure. But soft, friable lumps are unobjectionable in several cases, according to circumstances.

202. We have already referred incidentally to the fact that soluble salts may also be prepared by metathesis accompanied by precipitation, the precipitate being the bye-product. It is necessary in all such cases to carefully guard against contamination of the soluble product by the presence of an undue excess of one of the substances used as materials, and it may not infrequently be found advantageous to employ the exact proportions required

the hydrogen of the sulphuric acid which contributes the sulphate radical toward the formation of the copper sulphate and the two hydroxyls of the molecule of sulphuric acid which is reduced to SO_2 form, together, the two molecules of water.

When silver, mercury, copper, lead, or bismuth, is dissolved in nitric acid we have again a similar condition—reactions including both substitution and oxidation:

$$Bi+4HONO_2=Bi(NO_3)_3+2H_2O+NO,$$

or, to trace the interchanges more clearly:

$$Bi+HONO_2+3HNO_3=Bi(NO_3)_3+HOH+NO+H_2O.$$

When metallic oxides are dissolved in acids the bye-product is water formed by the hydrogen of the acid with the oxygen of the oxide:

$$ZnO+H_2SO_4=ZnSO_4+H_2O.$$

When metallic hydroxides are dissolved in acids the bye-product is again water, but twice as much water is then formed as when the oxide is used:

$$Zn(OH)_2+H_2SO_4=ZnSO_4+2H_2O.$$

When acids are saturated with metallic carbonates two bye-products are formed, namely water and carbon dioxide, because carbonic acid as soon as formed breaks up into H_2O and CO_2:

$$CaCO_3+2HNO_3=Ca(NO_3)_2+H_2O+CO_2.$$

208. The acids or other chemical solvents are said to be "neutralized" or "saturated" by the metal or metallic compound dissolved, and these expressions are used because the point of neutralization or saturation is frequently determined by the aid of "test-paper."

But in many cases the acid may be completely saturated by adding to it an excess of the metal or metallic compound. This can be safely done whenever there is no danger of the formation

of meta-compounds or basic salts, or other undesired products. It is safe to saturate sulphuric acid with zinc, iron, or copper, or hydrochloric acid with iron or zinc, or nitric acid with silver, lead or copper; but if salts of normal composition are sought nitric acid can not be saturated by mercury used in excess, or acetic acid by lead in excess.

209. In Chapter XIX, Vol. I, we have seen that the products of chemical solution [except gases which escape] must be soluble in the liquid in which the reactions take place; that acids do not dissolve metals and metallic compounds unless they form water-soluble salts with them [or salts soluble in the acid itself if the acid is strong or used in excess].

When the salt formed is not soluble in water but soluble in the acid, then a diluted acid can not be successfully employed unless used in considerable excess, and the result will then be a solution containing much free acid. Solutions of antimony chloride and of the nitrates of mercury and bismuth in water without the presence of much free acid can not be produced; but solutions of these compounds containing large proportions of the acids are made and are useful for certain purposes.

When the salt formed is quite soluble in water and in the dilute acid but insoluble in concentrated acid, then the undiluted acid does not dissolve the metal.

210. The action of the heavy metals upon the common inorganic acids. *Gold* and the *platinum metals* do not attack any acid, but they dissolve in nitrohydrochloric acid or *aqua regia*, forming chlorides with the free chlorine.

Aluminum decomposes hydrochloric acid, forming aluminum chloride and setting the hydrogen free. It does not attack other acids.

Antimony is oxidized by strong nitric acid to insoluble antimonous oxide. Other acids are not affected by antimony.

Tin decomposes strong nitric acid, forming metastannic acid. It also decomposes hydrochloric acid, forming stannous chloride. Sulphuric acid is not decomposed by tin.

Bismuth quickly decomposes nitric acid, forming bismuth nitrate, and it also attacks hot concentrated sulphuric acid, but not hydrochloric acid.

Silver attacks dilute nitric acid, forming silver nitrate; it also

decomposes hot concentrated sulphuric acid. It does not act upon hydrochloric acid.

Lead decomposes nitric acid, but scarcely affects hydrochloric and sulphuric acid.

Copper vigorously attacks nitric acid and also decomposes hot, strong sulphuric acid, but is not dissolved in hydrochloric acid or diluted sulphuric acid.

Nickel decomposes hydrochloric acid, sulphuric acid and nitric acid, forming nickelous salts.

Iron and *zinc* readily decompose the diluted acids.

211. *Hydrochloric acid* dissolves zinc, aluminum, iron, nickel and tin; it does not dissolve lead, copper, mercury, silver, gold, platinum, arsenic, antimony and bismuth.

Diluted sulphuric acid dissolves zinc, iron and nickel. Hydrogen is set free. But it does not dissolve aluminum, lead, copper, mercury, silver, gold, platinum, tin, arsenic antimony and bismuth.

Concentrated sulphuric acid dissolves copper, and, if *hot,* it is also attacked by mercury, silver and bismuth.

The acid not entering into the formation of the sulphate is reduced to SO_2.

Moderately dilute nitric acid, especially when warm, dissolves zinc, iron, nickel, lead, copper, mercury, silver, arsenic and bismuth. Arsenic is oxidized to arsenic acid; the other metals form nitrates. The acid not entering into the nitrate is reduced to NO, which oxidizes in the air to red vapors of N_2O_4, or NO_2, or both, according to the temperature.

Cold and very dilute nitric acid dissolves iron and zinc, forming ferrous nitrate or zinc nitrate together with ammonium nitrate.

Concentrated nitric acid is not attacked by iron, but dissolves lead, copper, mercury, silver, arsenic and bismuth. It is not affected by gold and platinum. It oxidizes tin to insoluble metastannic acid, and antimony to insoluble antimonous oxide.

212. The foregoing statements are not to be construed to mean that metals which are *not dissolved* by the acids named may not be superficially affected to a considerable degree. Diluted sulphuric acid *does* take up copper and form copper sulphate so that copper vessels are corroded by it; but the diluted acid dissolves the metal so slowly and to such a limited extent that we would not use diluted sulphuric acid for such a purpose, but con-

centrated acid instead. [Tin is not affected by sulphuric acid, but tinned iron or tin plate, however heavily coated with pure tin, is comparatively soon destroyed by not only very dilute sulphuric acid but even by boric acid solutions and by very weak acetic acid, probably because the tin coating is not so impervious that the iron is absolutely protected.]

213. *Metallic sulphides* are soluble in acids if their metals are such as perform strongly basic functions. Metallic sulphides corresponding to the acidic oxides are insoluble in dilute acids, but soluble in alkali solutions, as, for instance, the sulphides of arsenic and antimony. As zinc hydroxide is soluble in acids as well as in strong alkali solutions, its corresponding sulphide is also soluble in both acids and alkalies. Antimony sulphide dissolves in *very concentrated* hydrochloric acid, forming antimony trichloride and hydrogen sulphide; but is not soluble in dilute hydrochloric acid.

214. *Carbonates, sulphites* and *nitrites* of the metals are decomposed by all of the stronger acids because they yield gaseous bye-products. The acid-soluble sulphides also yield a gaseous bye-product, H_2S.

215. In chemical solution the salt or other soluble product formed by the reaction accumulates in the solution so as to gradually weaken the solvent and hinder if not entirely arrest the interaction. The products are *not* "removed from the arena of the chemical action" to as great an extent as in precipitation, and *the factors in chemical solution are not both of them liquids* in all cases. In most instances of chemical solution, only one of the factors is a liquid and the other usually a solid (rarely a gas). Hence the velocity of the reaction in chemical solution, while it may be great at first, may become so retarded later as to require acceleration by the application of heat. Zinc dissolves quite rapidly in dilute sulphuric acid until the solution contains much zinc sulphate and but little sulphuric acid is left; after that point has been reached the action is very slow, and, if it is desired to completely saturate all of the sulphuric acid, heat must be applied.

The usual method, when the bye-product is hydrogen, or carbonic acid, or any other gas, is to let the action go on without the aid of heat *until effervescence has ceased*, and then to apply

heat when the evolution of gas begins again and continues until saturation is completely effected.

216. *Neutralization* is effected in solutions by mixing acids and alkalies in the requisite proportions, adding either the acid to the alkali or the alkali to the acid. Acid salts are also neutralized by alkalies, and the alkali carbonates are employed almost as much as the alkali hydroxides. Whenever practicable the point of exact neutralization is determined by a color reagent, and the most common and useful reagent of this kind and for this purpose is **litmus,** which is generally employed in the form of *litmus paper.*

Litmus is a blue pigment prepared from certain lichens. It is turned red by acids; alkalies restore the blue color. White unsized paper dipped in a solution of litmus and dried is called litmus paper, or test-paper. The blue litmus paper is made from the unaltered solution of the pigment; the red litmus paper is made from a litmus solution to which just enough dilute hydrochloric acid has been added to turn its color red.

A liquid which turns blue litmus paper red is said to have an *acid reaction;* one that turns red litmus paper blue has an alkaline reaction; a liquid which does not change the color of either red or blue litmus paper is said to have a *neutral reaction* on test-paper or to be *neutral to test-paper.* The test is made by touching a small strip of the test-paper with the liquid, or the liquid with the test-paper.

In testing liquids with litmus paper it is necessary to guard against interferences which might vitiate the test or mislead the operator. It is quite possible, for instance, to conclude from the acid reaction of a liquid which contains a little carbonic acid that the salt in solution is an acid salt when in reality it may be perfectly neutral; this may occur when an acid is being neutralized with a carbonate. When a salt of acid reaction is being decomposed with ammonia, or an acid neutralized with it, the litmus paper may show an alkaline reaction produced by the ammonia vapor above the liquid while the liquid itself is still slightly acid or neutral.

217. When a strong acid is neutralized with a strong base, the salt formed is a normal salt. But normal salts formed by strong acids with weak bases have an acid reaction, and those formed by weak acids with strong bases have an alkaline reaction. Even

acid salts (salts still containing some of the replaceable hydrogen of the acid) may have a decidedly alkaline reaction as we find to be the case with the bicarbonates of potassium, sodium and ammonium; and so-called basic salts may have an acid reaction as is the case with solutions of subsulphate of iron. Several salts (notably sulphates and nitrates) of aluminum, iron, zinc, copper, and other metals have an acid reaction although their composition is normal. Even alum has an acid reaction.

218. When acids are *saturated* with the metals or with metallic oxides, hydroxides or carbonates the proportions employed of the materials are determined beforehand, even if an excess of the metal or metallic compound is to be used, and when salts of normal composition are to be prepared and the reaction on test-paper does not indicate the composition, the exact theoretical proportions are used.

219. The salts prepared by chemical solution, if crystallizable, are always recovered by crystallization because this method insures a product of definite composition and fine appearance if the process is well managed.

Many *salts of the heavy metals* crystallize most readily and satisfactorily and of perfectly normal composition from strongly acidulated solutions. Solutions of the sulphates are acidulated with sulphuric acid, those of nitrates with nitric acid, acetates with acetic acid, chlorides with hydrochloric acid.

Salts of the alkali metals, on the other hand, sometimes crystallize most readily from alkaline solutions. Solutions of potassium salts are rendered alkaline by the addition of a little carbonate or hydroxide of potassium, and solutions of sodium salts with sodium carbonate or hydroxide; solutions of ammonium salts are rendered alkaline with ammonia water.

But there are so many exceptions to these general statements that no inexperienced operator can be guided by them; special directions in individual cases are, therefore, given in all complete working-formulas.

220. Amorphous salts, and those that can not be advantageously produced in large crystals, are recovered from solutions obtained by chemical solution, by turbidation, granulation or evaporation to dryness, or by evaporation until a moist salt residue is obtained, which is then spread out on plates to dry.

221. In addition to the examples already given we shall men-

tion now a sufficient number of instances of chemical solution to show how varied such processes are: The production of phosphoric acid by dissolving phosphorus in nitric acid is an oxidation process and at the same time chemical solution; the production of Rochelle salt (tartrate of potassium and sodium) from potassium bitartrate (acid tartrate) and sodium carbonate is a process of chemical solution (neutralization); the production of ferrous iodide by dissolving iron and iodine together in water; the solution of zinc in hydrochloric acid to make zinc chloride; the solution of lead oxide in a solution of lead acetate to make solution of subacetate of lead; making solution of ferric citrate from ferric hydroxide, citric acid and water; the production of soluble ferric phosphate by dissolving precipitated ferric phosphate in a solution of citrate of ammonium, or by boiling sodium phosphate and solution of ferric citrate together; all of the processes by which the "scale salts" are made; the preparation of solution of potassium arsenite from arsenous oxide and potassium bicarbonate; the solution of ammonia gas in water to make the solution of ammonium hydroxide called ammonia water; the solution of carbon dioxide in water to produce a solution of carbonic acid; the preparation of solution of calcium hydroxide from calcium oxide (lime) and water; the formation of hydrogen dioxide by dissolving barium dioxide in water.

222. By the term **wet oxidation** I designate oxidation produced in the wet way—that is, in liquids. Wet oxidation is closely related to chemical solution, and in many cases is nothing else. As examples of wet oxidation we may mention: the method of turning ferrous salts into ferric by heating their solutions with nitric acid; making phosphoric acid from phosphorus with nitric acid; changing ferrous compounds into ferric by adding chlorine water, or by conducting a current of chlorine into the solutions containing them; producing potassium chlorate by the action of chlorine on potassium hydroxide in solution; producing potassium permanganate by the action of potassium chlorate and potassium hydroxide upon manganese dioxide, etc.

In pharmaceutical operations the most useful oxidizing agents are those that yield bye-products which can be easily eliminated or which are unobjectionable. Nitric acid is, therefore, much used.

In wet oxidation by nitric acid heat is generally applied to

facilitate the reaction and to expel the nitrogen oxide. The reducing agent is generally added in portions to the acid, and the red nitrous vapor is expelled after each addition before another portion of the reducing agent is added. Strong solutions favor rapid oxidation. But as the nitrous vapor is very suffocating and dangerous to inhale, these operations must be carried out under effective hoods, or in fume chambers, or out in the open air, where the vapors may be carried away from the operator and the air he must breathe.

223. Wet gas operations of various kinds are of common occurrence in laboratories, and may be appropriately mentioned here because they are intimately related to chemical solution and wet oxidation, frequently including both.

Hydrogen, nitrogen, chlorine, hydrogen sulphide, carbon dioxide, sulphur dioxide, nitrous acid, and various other gases are often produced in the wet way and most of them in no other way.

The *generators* used for this purpose may be ordinary wide-mouthed bottles, or flasks, or Woulff's bottles, or specially constructed apparatus such as shown in the illustrations.



Fig. 99. Kipp apparatus.

The Kipp apparatus, as shown here or modified for special purposes according to circumstances, is very useful for the production of gases as required for laboratory use. It may be employed for generating currents of hydrogen, oxygen, chlorine, hydrogen sulphide, carbon dioxide, and nitric oxide. For the evolution of these several gases the following named materials are placed in the bulbs: 1, for hydrogen the middle bulb is to contain granulated zinc and the others a mixture of 1 volume of sulphuric acid and 4 volumes of water; 2, for oxygen put pieces of compressed chlorinated lime in the middle bulb and a mixture of 100 volumes of hydrogen dioxide and 5 volumes of

nitric acid in the others; 3, for chlorine put compressed chlorinated lime in the middle bulb and a mixture of 7 volumes of hydrochloric



Fig. 100. Gas evolution apparatus.

acid and 5 volumes of water in the others; 4, for generating hydrogen sulphide, crushed ferrous sulphide is put in the middle bulb and equal volumes of hydrochloric acid and water in the others; 5,

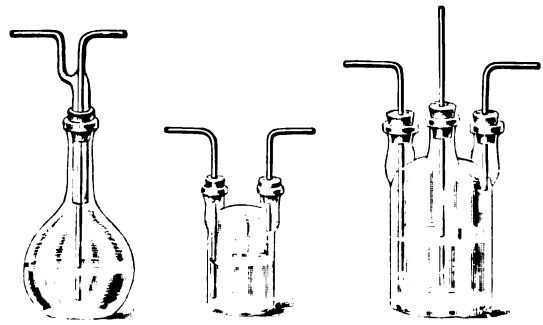


Fig. 101. Wash bottles. The last two are Woulff's bottles.

for the generation of carbon dioxide, broken marble is put in the middle bulb and equal volumes of hydrochloric acid and water

in the others; and 6, for nitric oxide use copper turnings and dilute nitric acid.

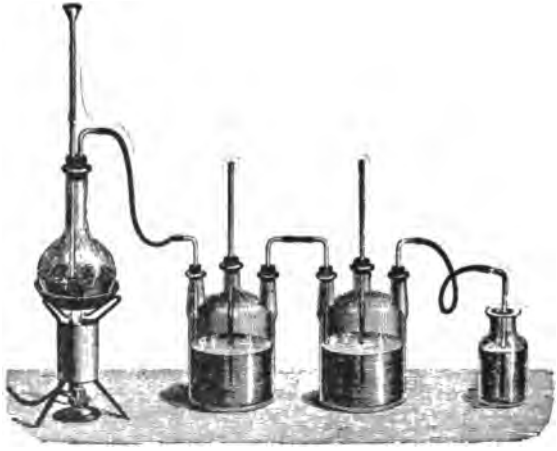


Fig. 102. Gas evolution apparatus.

224. Whenever it is necessary to *wash* the gaseous product to remove from it any other volatile substances which may accompany it out of the generator, the generator is connected with one or more *wash-bottles,* and the gas is, after having passed through the wash-water, conducted into the receiver where it is collected or dissolved in water. (See Fig. 101.)

Volatile acids are prepared in apparatus similarly constructed, and the whole process bears considerable resemblance to chemical distillation. The fittings necessary are practically the same as used for small distilling apparatus.

Several gas solutions are of considerable importance, as, for instance, solutions of chlorine, hydrogen sulphide, sulphurous acid, etc.

225. When gas solutions are made, the excess of gas passing out of the liquid contained in the receiving vessel should be conducted into an end-bottle for absorption into a fixing-solution if the escape of the gas into the room is objectionable. Thus the excess of chlorine or of nitric oxide may be conducted into a solution of sodium carbonate. (See Fig. 102.)

CHAPTER XIV.

THE USES OF UNFINISHED PRODUCTS—PURIFICATION OF CRUDE CHEMICALS—WHAT TO DO WITH DAMAGED PRODUCTS. PROFITABLE CHEMICAL WORK.

226. Well-trained pharmacists who have a moderately equipped laboratory and make use of it, find it decidedly profitable to employ unfinished products as materials. It is clearly wasteful and unworkmanlike to adopt an expensive method of making any preparation whenever a less expensive method leads to precisely the same result as to the quality of the finished article.

Chemical products in solid form, if they are water-soluble, are almost invariably obtained first in solution, and are afterwards recovered from their solutions by crystallization or some other process frequently involving the expenditure of considerable time and labor. If the solution contains the chemical compound of requisite degree of purity, and if all that remains to be done in order to finish the product is its recovery in solid form from the solution, then the cost of that operation is properly saved in every case where the solution itself can be directly employed. Perfectly pure and in every way satisfactory chemical products in a state of solution in water can be made without making the solution out of the finished solid substance, and it happens quite frequently that liquid preparations are to be made containing such chemical products.

When a large quantity of pyrophosphate of iron is required in the form of water-solution for the manufacture of some liquid preparation, the quantity needed is produced out of solution of ferric sulphate, sodium pyrophosphate, ammonia water and citric acid, or out of solutions of ferric citrate and sodium pyrophosphate, and the tedious and difficult process of turning the pyrophosphate of iron into scales is avoided.

If large quantities of solution of potassium iodide are required, it is cheaper and quite as satisfactory to make the solution out of iodine, iron and potassium carbonate instead of making it out of finished potassium iodide.

Nothing is gained by making a solution of sodium salicylate out of the finished salt instead of making it at less cost out of salicylic acid and sodium bicarbonate.

In the manufacture of elixirs, wines, and other liquid preparations containing pepsin, it is wasteful to use finished pepsin since a perfectly satisfactory solution of pepsin of ascertained strength can be made and the cost of scaling the pepsin saved.

Elixir of ammonium valerate can be produced more economically out of a solution made of valeric acid and ammonia-water than out of crystallized ammonium valerate, and the finished elixir is, of course, precisely the same whether prepared one way or the other.

Scarcely any class of preparations has taxed the capital and patience of the pharmacist more severely than the "elixirs." They represent an almost endless variety of combinations of comparatively few medicinal substances, among which are citrate of iron and ammonium, soluble phosphate of iron, soluble pyrophosphate of iron, quinine, strychnine, pepsin, citrate of bismuth and ammonium, etc. To keep in stock all of the different finished elixirs which are prescribed containing these substances requires very considerable capital. Moreover, it is practically impossible for the pharmacist to determine the age and actual condition of such preparations with any degree of certainty; he is practically reduced to the necessity of accepting them from the makers or dealers if they only seem to be right. And yet no medicinal preparations are more liable to rapid deterioration, so that they should always be recently prepared in order to be satisfactory. The skilled pharmacist, therefore, prepares the elixirs required to meet the demands upon his own establishment, as far as practicable, and, instead of keeping in stock a large variety of ready-made elixirs of which no one (not even the manufacturer after the product has left his laboratory) can possess or acquire sufficiently definite and complete knowledge, he may at less cost supply more reliable preparations by preparing them himself whenever required. This he can do by having on hand recently prepared solutions of the several medicinal substances named, these solutions being so made that they are any and all of them miscible with each other so that any combination whatever of them can without any difficulty be made whenever required by simply mixing the solutions in the requisite proportions with an ap-

propriately flavored and sweetened simple elixir of proper alcoholic strength to produce the desired result.

The foregoing illustrations are sufficient to demonstrate the value of technical education for the practice of pharmacy. But no person ignorant of pharmaceutical chemistry should undertake to do any such work because he could not know whether his results be right or wrong.

227. The purification of crude commercial chemicals is a not inconsiderable part of the profitable work of the pharmacist. Among the common crude inorganic chemicals which can be easily purified so as to be rendered fit for pharmaceutical and medicinal use we may mention, by way of illustration: boric acid, alum, ammonium chloride, copper sulphate, ferrous sulphate, lead acetate, lead nitrate, potassium chlorate, potassium nitrate, sodium carbonate, sodium thiosulphate, sodium phosphate, zinc sulphate. The cost of purification of these and many other substances is little or nothing beyond the value of the time and labor.

Brief general reference has been made in various places in this book to the purification of fusible solids contaminated with infusible impurities; the purification of volatile solids by sublimation to remove fixed impurities, and the removal of volatile impurities from fixed solids by ignition; the purification of crystallizable soluble substances by recrystallization, turbidation, granulation and physical precipitation; the removal of insoluble substances from soluble solids by solution and filtration; the separation of crystalloids and colloids by dialysis; the purification of liquids by filtration and by distillation; and the precipitation of foreign substances from solutions of various chemical compounds by substitution (as when zinc precipitates iron) or by metathesis.

Many individual examples of the purification of crude chemical products will be found in Part II of this volume.

228. Chemical products which have been damaged or altered, or mixed with other substances by accident, may generally be restored to their original condition, or rendered quite pure, or converted into some other valuable product, by simple methods.

A crystallized salt which has effloresced, or which has undergone aqueous fusion in hot weather; a hygroscopic or deliquescent substance which has absorbed moisture so that it is no longer in fit condition to be used; a ferrous salt which has

become partially oxidized to ferric; a compound of silver, gold or mercury, or any other valuable substance which has suffered partial decomposition from exposure to light; a salt accidentally dropped on the ground and thus rendered dirty; any substance ruined by admixture with any other substance; a solid containing minute fragments of glass from a broken container—none of these things cease to have some value, and any good pharmaceutical chemist should know how to repair damages of this kind or to reduce the loss to a minimum. Products which are found impure or otherwise unfit for the use for which they were intended may frequently be rendered fit for other uses.

The restoration of altered chemicals is further discussed in Chapter XV.

229. The pharmacist should further be able to prepare at any time, in an emergency or as a part of his regular occupation, almost any one of the many "small chemicals" which he is called upon to dispense, especially if he be located too far away from the usual sources of supply to be able to procure quickly whatever is wanted. He may often be able to make the preparation in much less time than is required to obtain it from any other source. Many chemical products which can be quickly and easily prepared may be so rarely called for that they are not easily procurable in any other way. But even the most common substances may be out of reach when most wanted and may be of such nature that it is easier to make them than to send for them. Finally, it has been frequently demonstrated in the laboratories of well equipped pharmaceutical schools that many of the newer and rarer chemical preparations, organic and inorganic, for which very high prices are charged by those who supply them, chiefly because they are new, can be very readily made by the pharmacist himself at far less cost.

230. A large number of pharmaceutical chemicals are of such a character that they can not be made to any greater advantage on a large scale than in very moderate quantities. All such products can be profitably made by any intelligent, well-trained pharmacist of proper business capacity. An extensive or generally well equipped laboratory is not necessary for this purpose.

Again, if his whole time is not occupied in other directions the ambitious educated pharmacist can hardly find any technical

work which will afford him greater satisfaction than the manufacturing of *one or more* carefully selected chemical products which he can make of unexceptionable quality and in considerable quantity. Organic as well as inorganic products belong to the list of chemicals which he can make it his special care to furnish, and any such product can doubtless be marketed through the regular distributing channels at a remunerative price.

CHAPTER XV.

THE PRESERVATION OF MEDICINAL SUBSTANCES.

231. The principal causes of alteration in chemical products, as these products are ordinarily kept, are light, heat, and contact with air or with moisture. Most of the changes which the chemicals undergo are slow, and in many cases they escape notice because not rendered conspicuous by alterations in color or other external properties. In some instances, however, the changes betray themselves by external signs.

232. Salts containing water of crystallization may effloresce and are then of unknown value, as the quantity of water lost can not be known until a chemical determination of the composition of the substance shall have been made, or the salt dried until it ceases to lose weight and attains a known constant composition. As the amount of water which may be lost by efflorescence is in many cases considerable, it is evident that effloresced substances are under no circumstances to be used for medicinal purposes; they should be recrystallized so as to be restored to their normal composition. Effloresced substances may be used in some cases for the production of other chemicals, but only by skilled pharmacists or chemists who can determine their exact value and who will then correctly compute the quantity required on the basis of that value.

Substances liable to effloresce are effectively protected from it by keeping them in tightly closed bottles in a cool place.

233. Salts which are readily water-soluble and contain a large proportion of water of crystallization may, in very hot weather, or when put in a hot place, undergo *aqueous fusion.* When cooled the salt, of course, solidifies again, and if contained in a tightly closed bottle it loses no water so that its composition is unaltered; but it forms a solid mass in the bottle from which it can not be removed except by dissolving it again either in its own water, as before, or, preferably, in more water. The salt may then be recrystallized, or the solution, made of definite strength, can be employed for any laboratory operation for which it may be required.

234. *Hygroscopic and deliquescent substances* may absorb moisture, and if the exact quantity of moisture they contain is not known they can no longer be used in the same proportion as if they were dry; and a moist, wet or liquefied salt may not be adapted for all the uses to which the dry salt is employed even if the percentage of moisture in it has been determined so that the correspondingly increased quantity required of the deliquesced salt is known.

The best plan is to dry the substance so as to restore it to its proper normal condition.

To prevent the absorption of moisture, all hygroscopic and deliquescent substances should be kept in tightly closed bottles in a dry place. The containers should not be too large in proportion to the rate of consumption of the contents.

235. *Substances of complex chemical structure,* or consisting of molecules composed of a large number of atoms, are generally less capable of resisting decomposition than are the simpler chemical compounds.

236. *Compounds of elements and compound radicals of indifferent chemical energy* decompose more readily than those formed by elements of decided and powerful positive or negative chemical polarity. Thus the compounds formed by nitrogen are often unstable, as are also those of gold, silver, mercury and several other weak positive metallic elements; but compounds formed by the alkali metals and the alkaline-earth metals are very stable, especially when the negative elements to which they are united are also powerful radicals. Salts of weak bases and of weak acids are usually unstable. Metals generally form weak acids when they form any acids at all. Elements which exhibit great power when exercising negative polarity (as, for instance, the halogens) generally form unstable compounds when they assume positive polarity.

237. *Compounds containing elements exercising an unusually high polarity-value capable of reduction* (in other words, powerful oxidizing agents), and *compounds containing elements exercising an unusually low polarity-value capable of augumentation* (in other words, powerful reducing agents), are less stable than compounds made up of elements exercising their normal polarity and the dominant valence which they exhibit when endowed with that polarity.

238. Light exerts a decided influence upon a large number of chemical compounds, inorganic as well as organic. This influence is in opposition to atomic attraction, and its immediate effect is, therefore, dissociation, accompanied by changes in the polarity-value of two or more of the component atoms of the molecule affected. In other words, light frequently induces reactions of oxidation and reduction in single chemical compounds as well as between two or more kinds of molecules in contact with each other.

The use of "instantaneous photography" demonstrates the fact that exposure to light for a period of 0.001 second is sufficient to decompose certain very sensitive or unstable chemical compounds. Most of the compounds of the so-called "noble metals" are decomposed by strong light, especially direct sunlight, and the dissociation wrought by its influence is frequently rapid.

Preparations of mercury, silver and gold must be carefully protected against light, and there are many other inorganic compounds and preparations which require similar protection. The "scale salts of iron" are all sensitive to light, and liquid preparations containing such scale salts *must all be kept in the dark* in order to prevent their deleterious alteration.

The pharmacopœias name specifically various medicinal substances which *must* be "kept in a dark place," or "kept in dark amber-colored bottles," or "protected against light;" but the list of substances directed to be thus protected should be considerably extended. The following substances should all be carefully protected against light:*

(Benzoic acid, phenol, *citric acid, gallic acid*), hydrobromic acid, hydrochloric acid, hydrocyanic acid, nitric acid, nitrohydrochloric acid, (*salicylic acid*), sulphurous acid, (*tannic acid, tartaric acid,* acetic ether), *ammonium benzoate,* ammonium iodide, *ammonium valerate,* (amyl nitrate), sulphurated antimony, (apomorphine hydrochloride), chlorine water, (*all aromatic*

*Although the scope of this book is limited to inorganic substances, I have included in this chapter the organic medicinal substances liable to change because some of the substances requiring protection are partly of organic and partly of inorganic origin, and because the sensitiveness of organic substances furnishes an altogether legitimate illustration of the chemical effects of light, heat, air, etc., upon matter in general. Moreover, this list may serve to call needed attention to the general neglect of this whole subject. The organic substances are those put in brackets.

waters), all silver compounds, iodide of arsenic, (*atropine and all its salts*), *gold and sodium chloride,* bismuth and ammonium citrate, (*caffeine and citrated caffeine,* chloral, chloroform, *chrysarobin, cinchonidine and all its salts, cinchonine and all its salts, cocaine and all its salts, elaterin,* eucalyptol), ferric chloride, all "scale-salts" of iron, saccharated ferrous iodide, *ferrous lactate,* ferric valerate, (*glycerite of phenol, glycerite of tannic acid*), *mercuric chloride,* calomel, mercuric cyanide, the mercurous and mercuric iodides, the yellow and the red oxide of mercury, yellow mercuric subsulphate, ammoniated mercury, *all other mercury compounds,* (*hydrastinine and all its salts, hyoscine and all its salts, hyoscyamine and all its salts,* iodoform), *solution of arsenic and mercuric iodide,* solution of ferric acetate, *solution of ferric chloride, solution of ferric citrate, solution of ferric nitrate,* solution of ferric subsulphate, solution of chlorinated soda, *lithium benzoate, lithium salicylate,* (methyl salicylate, *morphine and all its salts,* naphtol, paraldehyde), phosphorus, (physostigmine and all its salts, *pilocarpine and all its salts*), lead iodide, potassium permanganate, (pyrogallol, quinidine and all its salts, quinine and all its salts, *resin of podophyllum,* resorcin, *salol,* santonin, *sodium benzoate, sodium iodide,* sodium salicylate, *sodium paraphenolsulphonate,* (*sparteine and its salts,* spirit of nitrous ether), strontium iodide, (*strychnine and all its salts*), *syrup of hydriodic acid, syrup of ferrous iodide, syrup of the phosphates of iron quinine and strychnine,* (*veratrine*), *zinc iodide, zinc phosphide.*

[All *alkaloids and alkaloidal salts* should be protected against light because experience has shown that they are very generally sensitive to its decomposing effects. In fact *all organic chemicals* should be kept in the dark, or in dark amber-colored bottles.]

[*Volatile oils* and *oleoresins, aromatic waters* and *aromatic spirits* should all be kept in the dark.]

[*Fixed oils* and *fats, ointments, cerates* and *plasters* also require protection against light.]

[All *crude organic* (vegetable) *drugs,* and especially their *powders* must be carefully guarded against exposure to the damaging action of light, and, of course, also the *fluid extracts, extracts, tinctures, wines,* and *syrups* of all plant drugs. All *elixirs.*]

In the foregoing list the articles named in italics were not,

in the American Pharmacopœia of 1890, mentioned as requiring protection against light.

Dark amber-colored glass, and any earthenware which transmits no light whatever, constitute the most suitable material of which containers for medicinal substances can be made. Blue or purple glass, however dark, is the most mischievous—more so than perfectly colorless glass. But it is an excellent plan to put all *bottles* containing substances sensitive to light into tight wooden boxes, where they will not be reached by any light save when the boxes are opened. The bottles put in the boxes should be of dark amber-colored glass, tightly stoppered, and well wrapped in thick black or dark yellowish-brown paper.

239. Heat is in some cases nearly as damaging as light in its action upon compound matter. I refer now to such temperatures as may be met with under ordinary conditions in the officine or work-room of the pharmacist or in warerooms of dealers in drugs and chemicals. No matter how these rooms may be heated, the temperature is sometimes excessive, especially near the stove, registers, or radiators, and near the ceiling. In hot climates, and in very hot weather in moderate climates, the room temperature may often be so high that all medicinal substances alterable at temperatures above 15° or 20° are liable to suffer damage unless removed to the coolest place available. Finally, some portions of most stores and work-rooms are so exposed to the heat from the sun that substances liable to injury from light or heat, or both, should never be placed there.

By "a cool place" the pharmacopœias mean a place where the temperature never exceeds 15°, and does not fall below 10° C.

The substances which must be kept in a cool place are: 1, very volatile substances; 2, those liable to undergo aqueous fusion; 3, those liable to efflorescence; 4, substances prone to fermentation; 5, all fatty substances; 6, all volatile oils; and 7, unstable substances generally.

Among them we may specially mention: Strong acetic acid, strong nitric acid, ammonia water (these because they are concentrated solutions of volatile substances so that full bottles of them tightly stoppered and then kept in a very warm place may burst, or may prove dangerous to open on account of the rush of vapor from the contents); (oleic acid), sulphurous acid, (lard and benzoinated lard, lanolin, ether, acetic ether, all

alcohol), ammonium carbonate, ammonium valerate, (amyl nitrite, all ethers, all aromatic waters), chlorine water, hydrogen dioxide solutions, gold and sodium chloride, (benzin), bromine, chlorinated lime, (camphor, monobromated camphor), carbon disulphide, (all cerates, spermaceti, chloral, chloroform, all collodions, confections, copaiba, all plasters, eucalyptol), ferric chloride, all "scale salts" of iron, iron alum, saccharated iodide of iron, ferric valerate, (all valerates, iodoform), iodine, solution of ferric acetate, solution of magnesium citrate, solution of magnesium carbonate, solution of chlorinated soda, Vallet's mass, (honey, honey of rose, menthol, mucilage, all oleates, all oleoresins, all fixed oils and fats, all volatile oils, pancreatin, pepsin, paraldehyde), phosphorus, (pyroxylin), sodium salicylate, sodium sulphite, (spirit of ether, compound spirit of ether, spirit of nitrous ether), spirit of ammonia, (all aromatic spirits, all syrups, terebene, all ointments).

240. Contact with **air** is inimical to substances liable to oxidation, or which may take up carbon dioxide, or water from moist air, or which give up water of crystallization to dry air. Substances liable to undergo fermentation should also be protected against contact with air.

The most effective means of excluding air is to keep the substances requiring protection in small, completely filled, tightly closed containers of glass or porcelain. Glass-stoppered bottles are generally used and with very satisfactory results. In some cases, where the utmost care is necessary to prevent access of air and moisture, the glass-stoppers may be rubbed over with a uniform but very thin coating of pure petrolatum so that they make a smooth air-tight fit in the necks of the bottles.

When the pharmacopœias direct that any given substance shall be kept in "small bottles," the decision as to what size is most suitable is left to the judgment of the pharmacist. As long as the container is filled almost to the complete exclusion of air and remains tightly closed, the contents must remain effectively protected, no matter what may be the size of the vessel. But when the container is opened and the contents used, a portion at a time being removed, air is necessarily admitted each time. A "small bottle," then, is one that will hold no greater quantity than will certainly be consumed in a very short time, or before any portion can be injured; and in a few cases that means a quantity so small

that the whole of it is probably not much more than will be required at one time. In a pharmacy where syrup of ferrous iodide is so frequently dispensed that a pound bottle of it will be emptied in the course of a week or two, an "original bottle" of that size is not too large; but if 100 Gm of that syrup is more than will be dispensed in two weeks then an original bottle holding more than 100 Gm is *not* "a small bottle" within the meaning of the Pharmacopœia. It is, of course, far better to use stock containers somewhat too small for convenience than it is to use containers that are at all too large.

241. Impervious containers are nearly always necessary to the preservation of medicinal substances, and it is, therefore, bad practice to keep chemicals in containers of paper or wood. Any medicinal substance however inexpensive it may be, must be regarded as very valuable whenever it is used with benefit as a remedy, and for that purpose it must be at all times in proper condition. If in order to preserve a given medicinal agent it should be necessary to use a container the cost of which is greater than that of the medicinal substance itself, it is nevertheless clearly inadmissible to use a cheaper container. There is no valid excuse for any deviation from that principle. In many pharmacies it is the uniform practice to dispense all medicines so far as practicable in glass stoppered bottles, and corks are not used. This may seem to occasion in some cases an unnecessary additional expense to the consumer, but the proportion of cases in which it is decidedly best from the point of view of the welfare of the patient is so great and the additional cost is so trifling that this practice deserves universal adoption as one of the unwritten laws of correct pharmacy.

When manufacturers of chemical products put up their goods in paper cartoons or wooden boxes they generally do so under protest in the form of a printed label which declares that the practice is wrong, and that the customer who insists upon it in order to save the cost of a proper container must take the responsibility for the consequences.

Corks and rubber stoppers are very unsatisfactory and objectionable. Glass-stoppered bottles are now so inexpensive that they ought to be generally if not exclusively used. When corks are used the tops of the containers are usually covered with wax

or paraffin to close up the worm holes and other defects in the corks.

242. Among the substances which should be kept in small, tightly closed bottles (glass-stoppered bottles) are:

Hydrocyanic acid, sulphurous acid, (amyl nitrate, apomorphine hydrochlorate), chlorine water, solution of hydrogen dioxide, gold and sodium chloride (chloral, chloroform, the collodions, elaterin), saccharated carbonate of iron, saccharated iodide of iron, ferrous lactate, ferrous sulphate, ferric valerate, (glycerite of tannic acid), mercurous iodide, mercuric iodide, yellow mercuric oxide, yellow subsulphate of mercury, ammoniated mercury, (hyoscine and its salts, hyoscyamine and its salts), magnesia, Vallet's mass, (morphine acetate, the oleates, all volatile oils, fixed oils), phosphorated oil, (physostigmine and its salts, pilocarpine and its salts), lead acetate, potassium hydroxide, potassium acetate, potassium cyanide, sodium bisulphite, sodium iodide, (spirit of nitrous ether), syrup of hydriodic acid, syrup of ferrous iodide, the bromide, chloride, iodide and phosphide of zinc.

243. All strong acids, solutions of the alkalies, the solid alkalies, bromine, iodine, and other substances of a corrosive nature or having a destructive action on corks, must, of course, be kept in glass-stoppered bottles.

244. Moisture affects all hygroscopic and deliquescent substances. All such substances must, therefore, be kept in perfectly dry, small, tightly closed (well made glass-stoppered) bottles. Potassium hydroxide, potassium acetate, carbonate, and cyanide, the halides of zinc, ferric chloride, and several other substances are of this class.

CHAPTER XVI.

SOLUBILITIES OF INORGANIC CHEMICAL COMPOUNDS IN WATER AND IN ALCOHOL.

245. Few if any students or even experienced pharmacists are able to keep in their memories the solubilities of individual substances. But any one having a moderately retentive memory may easily learn and remember certain general statements concerning the solubilities or want of solubility of whole groups of compounds, together with the exceptions to those general statements. Such a memorization of general rules relative to the solubilities of inorganic chemical compounds is of such great value to both student and practitioner of pharmacy that a chapter specially devoted to this subject is here presented.

For this purpose we shall group the metallic compounds in two different ways, as has already been done in this book for other purposes, namely: 1, according to the metals; and 2, according to the negative radicals with which the metals are combined.

SOLUBILITIES OF COMMON METALLIC COMPOUNDS GROUPED ACCORDING TO THE METALS IN THEM.

246. Potassium compounds. All are water-soluble. Most of them are readily soluble.

Deliquescent are the—

Hydroxide, carbonate, cyanide, phosphate, hypophosphite, and acetate and sulphurated potassa.

Readily soluble are—

Bicarbonate, chloride, bromide, iodide, ferricyanide, ferrocyanide, nitrate, tartrate, citrate, salicylate and benzoate, and Rochelle salt.

Less readily soluble are—

Sulphate, in 9.5 parts of water at 15°.

Dichromate, in 10. parts of water at 15°.

Permanganate, in 16. parts of water at 15°.

Chlorate, in 16.7 parts of water at 15°.

Very sparingly soluble—

Cream of tartar, in 201 parts of water at 15°.

Nearly insoluble—

Potassium-platinum chloride.

247. Sodium compounds. All are soluble except the antimonite, which is nearly insoluble.

Very *freely soluble* are—

Hydroxide, carbonate, chloride, bromide, iodide, chlorate, sulphate, sulphite, bisulphite, thiosulphate ("hyposulphite"), nitrate, nitrite, phosphate, hypophosphite, arsenate, paraphenolsulphate, acetate, tartrate, citrate, valerate, salicylate and benzoate and Rochelle salt.

Less readily soluble—

Bicarbonate, in 11.3 parts of water at 15°.

Pyrophosphate, in 12. parts of water at 15°.

Tetraborate (borax), in 16. parts of water at 15°.

Bitartrate, sparingly.

248. Lithium compounds. All freely soluble in water except the carbonate, which dissolves in 80 parts of water at 15°.

249. Ammonium compounds. All officinal ammonium compounds are readily water-soluble, the least soluble being the benzoate and the carbonate, which are each soluble in 5 parts of water at 15°.

250. Barium salts. Nitrate, chloride, bromide, iodide, sulphide and acetate are *readily soluble.*

Hydroxide soluble in 20 parts of water.

Insoluble are—

Carbonate, phosphate, sulphate and oxalate.

251. Strontium salts. Chloride, bromide and iodide are *deliquescent.*

The acetate, lactate and nitrate are *readily soluble.*

The hydroxide is comparatively *sparingly soluble.*

Insoluble are—

Carbonate, phosphate, sulphate and oxalate.

252. Calcium compounds.

Deliquescent are—

Chloride, bromide, iodide.

Readily soluble—

Nitrate, hypophosphite, sulphite, acetate, lactate, and sulphurated lime.

Sparingly soluble—
Hydroxide, in from 600 to 700 parts of water at 15°.
Sulphate, in about 300 to 400 parts of water at 15°.
Insoluble—
Carbonate, oxalate, phosphate.

253. Magnesium compounds. *Readily soluble* are the—
Chloride, bromide, iodide, nitrate, sulphate, acetate, lactate and the acid citrate.
Insoluble are—
Oxide, hydroxide, carbonate, oxalate, phosphate.

254. Zinc compounds.
Deliquescent are—
Chloride, bromide and iodide.
Readily soluble are—
Sulphate, nitrate, acetate, lactate and paraphenolsulphonate.
Less readily soluble—
The valerate.
Insoluble—
Oxide, sulphide, phosphide, hydroxide, carbonate, oxalate, phosphate, oleate.

255. Cadmium compounds.
Soluble are the—
Chloride, bromide, iodide, nitrate and sulphate.
Insoluble are the—
Oxide, hydroxide, sulphide, carbonate, oxalate, phosphate.

256. Aluminum compounds.
Readily soluble are—
Chloride, bromide, iodide, nitrate, sulphate, acetate, potash-alum, and ammonia alum.
Insoluble are the—
Oxide and hydroxide.

257. Cerium compounds.
Soluble are the chloride, nitrate and sulphate.
Insoluble are the oxide, hydroxide, carbonate and oxalate.

258. Cobalt compounds.
The *cobaltous salts* are *deliquescent.*
Insoluble are the—
Oxides, hydroxides and sulphides.

259. Nickel compounds.
Nickelous sulphate and nickelous chloride are *soluble.*

Oxides, hydroxides and sulphide are *insoluble.*

260. Iron compounds.

Very *readily water-soluble* are—

Ferrous chloride, bromide and iodide.

Ferrous sulphate.

Ferric chloride and bromide.

Ferric nitrate, subsulphate, sulphate, acetate, and citrate, iron alum.

The *Scale-Salts* of iron are all *freely soluble.*

Less soluble—

Ferrous lactate is soluble in 40 parts of water at 15°.

The *Insoluble Iron Compounds* are—

Ferrous and ferric—

Oxides, hydroxides, sulphides, carbonates, oxalates, phosphates, pyrophosphates, metaphosphates, hypophosphites.

261. Chromium compounds.

Water-soluble are—

Chlorides, chromium sulphate, chromic anhydride (so-called "chromic acid"), potassium chromate, potassium dichromate, and chrome alum.

Insoluble are—

Oxide, hydroxide.

262. Manganese compounds.

Soluble are—

Manganous chloride, bromide, iodide, nitrate and sulphate. Also potassium manganate and permanganate.

Insoluble are—

The oxides, hydroxide, carbonate, oxalate, phosphate, sulphide.

263. Lead compounds.

The only water-soluble lead compounds are—

Nitrate, acetate and subacetate.

264. Copper compounds.

The only water-soluble cupric compounds are—

Chloride, nitrate, sulphate and acetate.

265. Mercury compounds.

All mercurous compounds are insoluble in water; but mercurous nitrate is soluble in a mixture of water and nitric acid.

The only *water-soluble mercuric compounds* are—

Chloride (1+16), bromide, acetate and cyanide. But mercuric nitrate is soluble in a mixture of nitric acid and water

266. Silver compounds.

The only *water-soluble* silver salts are the nitrate and acetate.

267. Gold. The only water-soluble gold compound in use is the trichloride.

268. Bismuth compounds. *The only water-soluble bismuth compound* is the citrate of bismuth and ammonium.

But normal bismuth nitrate, which is decomposed by water, is soluble in glycerin and also in glacial acetic acid. It is also soluble in a mixture of nitric acid and water.

269. Antimony compounds.

The only water-soluble antimony compound is tartrate of antimonyl and potassium (tartar emetic).

But chloride of antimony is soluble in a mixture of hydrochloric acid and water.

270. Arsenical compounds.

Sodium arsenate is *readily soluble.*

The arsenite of potassium is also soluble.

Iodide of arsenic is soluble.

Arsenous acid is sparingly soluble.

SOLUBILITIES OF COMMON INORGANIC CHEMICAL COMPOUNDS GROUPED ACCORDING TO THE NEGATIVE RADICALS OF THEIR MOLECULES.

271. Oxides. No metallic oxides are water-soluble except chromic anhydride and other oxides that react with the water to form either acids or bases; those dissolve by "chemical solution."

272. Hydroxides. The only *freely water-soluble* metallic hydroxides are those of the alkali metals.

The hydroxides of barium, strontium and calcium are comparatively sparingly or very *sparingly soluble.*

All other metallic hydroxides are *insoluble.*

273. Chlorides. All metallic chlorides are water-soluble except those of silver and lead, mercurous chloride, and the chloride of antimony, which is decomposed by water but soluble in a mixture of hydrochloric acid and water.

Deliquescent chlorides are those of—

Calcium, zinc; also ferric chloride and platinic chloride.

Readily soluble are the chlorides of—

Potassium, sodium, lithium, ammonium, barium, strontium, magnesium, aluminum, and gold.

Less readily soluble is—

Mercuric chloride (1+16).

Insoluble—

Silver chloride and mercurous chloride.

Nearly insoluble—

Lead chloride.

Decomposed by water—

Antimony trichloride.

274. Bromides.

Readily soluble are the bromides of—

Potassium, sodium, lithium, ammonium, barium, strontium, calcium, magnesium, zinc, aluminum, and ferrous and ferric bromide.

Soluble are—

Mercuric bromide and bromide of gold.

Insoluble—

Bromides of silver and lead.

Mercurous bromide.

275. Iodides.

Freely soluble are the iodides of—

Potassium, sodium, lithium, ammonium, barium, strontium, calcium, magnesium, zinc, cadmium, ferrous iodide, manganous iodide, and arsenous iodide.

Insoluble are the iodides of—

Silver, lead, mercury (mercurous and mercuric).

276. Cyanides. Those of the alkali metals are *freely soluble.*

Mercuric cyanide is *soluble.*

Cyanide of silver is *insoluble.*

277. Ferrocyanides and ferricyanides of the *alkali metals* are water-*soluble.*

Those of the *heavy metals* are all *insoluble.*

278. Sulphides. Those of the alkali metals and the alkaline-earth metals are *freely water-soluble.*

All sulphides of the *heavy metals* are *insoluble.*

279. Hypochlorites of the alkali metals and alkaline-earth metals are *soluble.*

280. Chlorates of potassium and sodium are soluble.

281. Sulphites. Those of potassium and sodium are *readily soluble;* those of calcium and magnesium less readily *soluble.*

282. Sulphates. All metallic sulphates are soluble except those of barium, strontium, calcium, lead and mercury.

Readily soluble sulphates are those of—

Sodium, ammonium, aluminum, and ferric sulphate. Also the alums.

Soluble are also the sulphates of—

Potassium, lithium, magnesium, zinc, ferrous sulphate, manganous sulphate, copper sulphate.

Very sparingly soluble—

Calcium sulphate.

Insoluble are the sulphates of—

Barium, strontium and lead.

Decomposed by water is the sulphate of mercury (mercuric).

283. Thiosulphates of potassium and sodium are *freely soluble.*

284. Sulphurated potassa and **sulphurated lime** are very freely soluble.

285. Nitrates. All are water-soluble except those of mercury and bismuth, which are decomposed by water.

286. Nitrites of alkali metals are soluble.

287. Phosphates, pyrophosphates and metaphosphates. The only water-soluble phosphates are those of the alkali metals and ammonium.

But some phosphates of the heavy metals and also the phosphates of the alkaline-earth metals and magnesium are soluble *in phosphoric acid.*

Orthophosphates of iron are soluble in orthophosphoric acid but insoluble in pyrophosphoric or in metaphosphoric acid.

Pyrophosphates and metaphosphates of iron are insoluble in orthophosphoric acid but soluble in metaphosphoric acid.

288. Hypophosphites. Those of the alkali metals and of calcium are water-soluble. Those of the heavy metals are insoluble.

289. Carbonates. The only water-soluble carbonates are those of potassium sodium and ammonium. That of lithium is sparingly soluble.

290. Borates. Sodium tetraborate (or borax) is soluble.

291. Metallic **acetates** are all water-soluble.

292. Valerates. Only those of potassium, sodium, lithium and ammonium are soluble.

293. Oxalates. Only those of the alkali metals and ammonium are soluble.

294. Tartrates. The normal tartrates of the alkali metals and ammonium are *soluble;* their bitartrates are *sparingly soluble.*

Tartrate of ferryl and potassium. Tartrate of ferryl and ammonium, and tartrate of antimonyl and potassium are *soluble.*

295. Citrates. Those of the alkali metals, ammonium and of iron are *soluble.*

Magnesium citrate is soluble in water containing much citric acid.

Bismuth citrate is *insoluble;* but citrate of bismuth and ammonium is soluble.

296. Lactates. Those of the alkali metals, calcium, strontium, magnesium, zinc and iron are water-soluble.

297. Salicylates. Those of the alkali metals are alone soluble.

298. Phenolsulphonates. Those of the alkali metals, barium, calcium and zinc are water-soluble.

299. Benzoates. Those of the alkali metals, ammonium and calcium, are water-soluble.

300. Oleates. Only the soaps are water-soluble.

301. The student will find it useful to specially memorize the following:

All of the officinal compounds of potassium, sodium and ammonium are water-soluble; but cream of tartar is only very sparingly soluble.

The hydroxides of potassium sodium and ammonium are freely soluble. [Lithium hydroxide is soluble in about 14 parts of water; barium hydroxide ($Ba(OH)_2.8H_2O$) dissolves in 20 parts of water; strontium hydroxide ($Sr(OH)_2.8H_2O$) is soluble in 50 parts; calcium hydroxide ($Ca(OH)_2$) is soluble in about 640 parts of water.]

The oxides, hydroxides, sulphides, carbonates, oxalates, phosphates (incl. pyrophosphates and metaphosphates), hypophosphites, arsenates and arsenites, salicylates, benzoates and oleates of the *heavy metals* are all insoluble.

ALCOHOL SOLUBILITIES OF COMMON CHEMICALS.

302. A very large proportion of the inorganic chemical compounds are insoluble in **alcohol,** and especially those that contain much water of crystallization. Inorganic chemical compounds which are insoluble in water are also, with scarcely any exceptions, insoluble in alcohol; but a large number of water-soluble inorganic chemicals are insoluble in alcohol.

303. Very soluble (in less than 5 parts of alcohol): [Benzoic, citric, salicylic, tannic and tartaric *acids.*]

Hydroxides of potassium, sodium and ammonium.

Chlorides of magnesium, zinc, iron (both ferrous and ferric), and mercuric chloride.

Bromides of lithium, barium, strontium, calcium, magnesium, and zinc.

Iodides of lithium, sodium, barium, calcium, magnesium and zinc.

Acetate of potassium.

Valerates of potassium, sodium, ammonium and iron (ferric).

Salicylates of potassium, sodium, lithium and ammonium.

Ferric *sulphate.*

304. Soluble in from 6 to 30 parts of alcohol:

Iodine (1+10).

Boric *acid* (1+15).

Chlorides of lithium (1+10), strontium (1+6), calcium (1+8).

Bromides of sodium (1+13), ammonium (1+30), and mercuric bromide.

Iodides of potassium (1+18), ammonium (1+9), strontium, and cadmium.

Mercuric *cyanide* (1+15).

Nitrate of ammonium (1+20).

Hypophosphite of potassium (1+7.3) and sodium (1+30).

Acetate of sodium (1+30) and lead (1+21).

Lactate of strontium.

Benzoate of lithium (1+12) and ammonium (1+28).

Sparingly soluble (in from 36 to 200 parts):

Bromide of potassium (1+200).

Iodide, mercuric (1+130).

Chlorate, sodium (1+100).

Nitrate, sodium (1+100).
Bisulphite, sodium (1+72).
Phenolsulphonate, sodium (1+1·32).
Acetate, zinc (1+36).
Valerate, zinc (1+40).
Benzoate, sodium (1+45).

305. Insoluble, or nearly so, in alcohol:

All metallic *carbonates, oxalates, phosphates, pyrophosphates, metaphosphates, arsenates, arsenites, citrates* and *tartrates.*

All metallic *sulphates* except ferric sulphate.

All the "scale-salts" of iron.

The—

Chlorides of potassium, sodium and ammonium, and mercurous chloride.

Iodide of lead and mercurous iodide.

Cyanide of potassium.

Nitrates of potassium, lead, copper and mercury.

Nitrate of sodium.

Chlorate of potassium.

Sulphite of sodium.

Thiosulphate of sodium.

Borax.

Potassium dichromate.

Potassium ferrocyanide and ferricyanide.

Ammoniated mercury.

Ferrous lactate.

CHAPTER XVII.

THE DENSITY OF SOLIDS AND LIQUIDS. THE MOHR-WESTPHAL BALANCE. HYDROMETERS, PYCNOMETERS, ETC.

306. It is very frequently necessary in laboratory operations to take the density of various liquids. As this happens most frequently in the process of concentrating solutions by evaporation preparatory to crystallization, and as density is a convenient guide to the approximate strength of solutions, we shall sufficiently discuss this subject before we describe evaporation and crystallization.

But the student must consult special works on weights and measures, specific weight, balances, and other apparatus and instruments, to learn the principles involved in their construction and the details of their care and use.

307. The **density** of any substance is the relation of its mass to its volume. It is expressed in units representing the density of some known substance adopted as the medium of comparison. The densities of solids and liquids are thus expressed by numbers, the significance of which is understood from the fact that the value of each unit is the density of water. The densities of gases or vapors are compared to and expressed in units of the density of hydrogen.

The term *specific gravity* is most commonly employed to express density; and the term *specific weight* is also used with the same meaning.

Accordingly, the density, specific weight, or specific gravity of water, as these terms are commonly employed, is 1; and the density, specific weight or specific gravity of hydrogen is also 1. But water is about 11,160 times as heavy as hydrogen, so that the density of water expressed in units of the density of hydrogen is 11,160; and the density of hydrogen expressed in units of the density of water is $\frac{1}{11,160}$ or 0.0000896. These numbers are so inconvenient as to forbid the employment of the water unit to express the densities of gases or of the hydrogen unit to express the densities of solids and liquids.

The density of air was formerly employed (and is still used to a considerable extent) for expressing the densities of gases; but it can not be used as the unit of expression in stating the densities of solids and liquids, and there are strong reasons in favor of the settled preference for hydrogen as against air as the medium of comparison of the densities of gases.

Air is about 14.43 times as heavy as the same volume of hydrogen, so that the density of dry air in hydrogen units is about 14.43 and the density of hydrogen in units of the density of dry air is about 0.0693.

308. Since inorganic pharmaceutical chemical products are solids and liquids, it follows that the unit of density, or of specific gravity, or specific weight, most important to the laborant in this field of work is the density of water. The form of expression of the density of any given solid or liquid is, therefore, a number which shows how many times the density of water (the unit) is contained in the density of that given solid or liquid. Thus the statement that the density, or specific weight, or specific gravity of any given solid is 8.000 means that a given volume of that solid weighs 8 times as much as the same volume of water; the sp. gr. of any liquid is 1.250 if a given volume of it weighs 1.250 times as much as an equal volume of water; the sp. w. of castor oil is 0.960 because one liter of it weighs 960 Grams; and one liter of the official "solution of subacetate of lead" weighs about 2.100 kilograms because its density is 2.100.

For the sake of uniformity and precision the specific weights of all liquids are expressed in numbers carried out to the third decimal, and in a few cases, where circumstances require it, even four decimals are given.

309. The densities of all substances are affected by temperature, pressure, buoyancy, and other conditions. It is, therefore, necessary to know the *conditions* to which any expression of density refers. For all ordinary purposes the standard temperature of 15° C. is almost universally adopted, and most of the pharmacopœias of recent date state the specific gravities of liquids at 15° C. in numbers which refer to the density of water *at the same temperature* as the unit of expression. But some pharmacopœias give the specific gravities of liquids at 15° C. in units referring to the density of water at 4° C.

The buoyancy and atmospheric pressure are generally ignored

in the determination and expression of specific gravities for industrial purposes and in the pharmacopœial tests; but whenever scientific accuracy is necessary the standard unit is the density of water at 4° C. *in vacuo,* and the true density or specific weight of any given liquid or solid is the number which shows how many times the actual mass (or weight *in vacuo*) of a given volume of water at 4° C. is contained in the actual mass (or weight *in vacuo*) of the same volume of that given liquid or solid at the same temperature.

The Swiss Pharmacopœia gives the *true densities* (specific weights derived from the true masses of the respective liquids at 4° C.); but the American Pharmacopœia, in common with the great majority of other pharmacopœias and other technical working manuals, gives the *apparent densities* (specific weights derived from weights in air at 15° C.) of the liquids described in it.

The difference between the true *mass* of any liquid or solid and its *weight in air* is the true weight or mass of the same volume of air.

310. The **law of archimedes,** or the *law of buoyancy,* may be expressed as follows: *Any body of matter immersed in any fluid* [and by "fluid" is here meant any liquid or gas] *is pushed upward by the gravitation thereof with a force exactly measured by the weight of its own volume of that fluid.*

A cubic-centimeter of lead resting upon the table is immersed in air; therefore it does not press upon the table with a force equal to the mass or true weight (weight *in vacuo*) of one cubic-centimeter of lead, but with a force less than the mass of the metal by just the weight of one cubic-centimeter of air. The mass of one cubic-decimeter of lead is 11,400 Grams; that of one cubic-decimeter of water is 1,000 Grams; that of one cubic-decimeter of dry air is 1.29303 Grams; that of one cubic-decimeter of mercury is 13,600 Grams; and that of one cubic-decimeter of hydrogen 0.0896 Grams. Consequently it follows from the law of buoyancy that one cubic-decimeter of lead exerts a pressure of 11,400 Grams upon its support in a vacuum, but a pressure of only 10,400 Grams on the bottom of a vessel of water; when weighed in air it would seem to weigh about 11,399 Gm, in hydrogen almost as much as in a vacuum, and, when suspended in water, only about 10,400 Grams; and if thrown into mercury the lead must float for it can sink down into the mer-

cury only far enough to displace 11,400 Gm (its own weight) of the mercury.

311. Buoyancy is the effect of gravitation, for mass is measured by gravitation, density is the relation of mass to volume, and a body of greater density is, therefore, a body acted upon by a greater force of gravity. Lead bullets find their way to the bottom of a basketful of beans because the lead is attracted toward the center of the earth with much greater force than the bean substance. Very heavy ores or other minerals are, therefore, sometimes separated from other substances on this principle, as grain is separated from the chaff, or as the heavy seeds are separated from the light spongy pulp of dried colocynth fruit.

Hydrometers and various other instruments designed to determine the specific weights or densities of liquids and solids are constructed on the principle of the law of buoyancy.

312. The density of a solid heavier than water and unaffected by it may be found as follows: weigh the solid first in air and then suspended in water; the difference is the weight of the same volume of water, and the specific weight of the solid is the quotient found by dividing its weight in air by the weight of the same volume of water

Should the solid be water-soluble it may be weighed in some liquid by which it is not affected and the density of which is known. Let us suppose that oil of turpentine is decided upon as fulfilling the necessary conditions. The difference between the weight of the solid in the air and its weight when suspended in oil of turpentine is, of course, the weight of the same volume of that oil, and the weight of the same volume of water is the quotient obtained by dividing the weight of the oil by its specific weight. We then divide the weight of the solid in the air by the weight of the same volume of water.

Solids which are lighter than water may be weighed in lighter liquids, or they may be weighted down with heavy substances and thus weighed in water after which the weight of the water displaced by the heavier solid is deducted from the weight of the water displaced by both solids to find the weight of the water displaced by the lighter solid.

But the volume of any solid can, of course, be readily found by submerging it in any lighter liquid (by which it is unaffected) in a graduated cylinder, for the rise of the level of the liquid

must correspond exactly to the volume of the solid. The densities of powders are usually found by this means.

The densities of lard and other soft fats and substances which can not be weighed suspended in liquids may be found by placing them in a heavier liquid and then gradually adding a lighter liquid until the density of the liquid is identical with that of the solid which may be known to be the case when the solid neither rises to the surface nor sinks to the bottom of the liquid, but may be made to remain suspended at any point in it; or the solid may be placed in a lighter liquid after which a heavier liquid is added until the densities of solid and liquid are equal, as described. The solid must of course be insoluble in both liquids, and unaffected by either of them, and the liquids must be miscible with each other in all proportions.

313. The densities of liquids also may be found by weighing solids in them, since the apparent loss of weight of any solid when weighed in any liquid (or the difference between the weight of the solid in the air and its weight when suspended in the liquid) must be the weight of the same volume of that liquid. As the weight of 10 milliliters of water is 10 Gm, it follows that the weight of a piece of glass measuring 10 cubic-centimeters must be 10 Gm less in water than in air, and it must be 9 Gm less in any liquid of the sp. gr. 0.900, and 12 Gm less in any liquid of the sp. gr. 1.200.

Thus, a piece of glass displacing 10 milliliters of water must lose 10 Gm when weighed in water and its loss of weight in any other liquid, expressed in Gm, when divided by 10, must give a quotient coincident with the sp. w. of that other liquid.

314. Hydrostatic balances are specially constructed balances for determining the specific weights of liquids and solids by weighing solids in liquids.

The balances of Mohr and Westphal are the best hydrostatic balances, and the most accurate determinations of specific weight may be obtained by the Mohr-Westphal balances (Fig. 103).

The solid to be weighed in the liquid is a short glass thermometer suspended from the end of the beam by a platinum wire. This thermometer is so constructed that when it is suspended in air the balance is in perfect equilibrium. When the thermometer is suspended in any liquid in the cylinder, as shown in the cut, it is buoyed up by the gravitation of the liquid so that

weights are necessary to restore equilibrium; these weights show the weight of the liquid displaced. The weights shown in the illustration are "riders." Each smaller rider is just one-tenth the weight of the next larger, except the counterpoise which equals in weight the water displaced and which, in the cut, is attached to the same hook that supports the thermometer.

As the product obtained by multiplying the power by its distance from the fulcrum is equal to the product obtained by mul-



Fig. 103. The Mohr Westphal balance.

tiplying the load by its distance from the fulcrum, it follows that when the distance of the power from the fulcrum is the same as the distance of the load from it, the power and the load must be equal. The beam of the balance is a "lever of the first kind," having the fulcrum between the power and the load. Hence, when the longer arm of the beam or lever is divided into ten equal spaces is must follow that any rider placed in any given

notch on the beam must weigh one-tenth more in that notch than it does in the next notch nearer the fulcrum. It is, therefore, immaterial whether or not the absolute weight of each rider is known if the relative weights are only correct. It will be seen that there are five riders. The weights of the two largest riders are equal; the third in size weighs one-tenth as much, the fourth weighs one one-hundredth, and the fifth and smallest weighs one one-thousandth as much as the largest.

When the riders have been so placed that the equilibrium of the balance is perfect the specific weight of the liquid can be read off from the numbers on the graduated beam. Should the liquid be water the counterpoise alone will suffice to establish equilibrium by placing it on the hook to which the platinum wire is attached; the counterpoise is, of course, always used when the liquid is heavier than water, but omitted when the liquid is lighter. The manner of reading the specific weight indicated by the riders is sufficiently illustrated by the examples shown in Fig. 104.

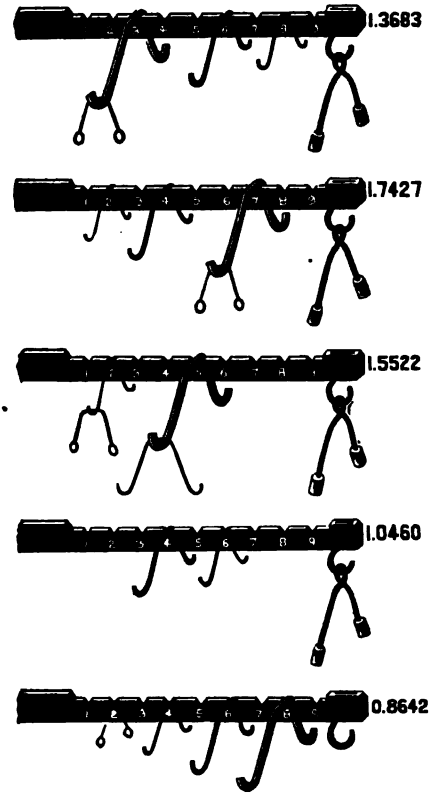


Fig. 104. How to read the indications of the riders.

315. A simple hydrostatic balance may be extemporized by replacing one of the stirrups of an ordinary good simple lever equal-armed balance by another and much shorter stirrup, soldering a hook to the bottom of the pan placed on that short stirrup, and restoring the equipoise by using enough solder to effect that result. A plummet of glass is then suspended by a platinum

wire to the hook under the pan, and may be weighed in any liquid, the sum of the weights necessary to overcome the force of the buoyancy being the weight of the liquid displaced. The weight of the water displaced by the plummet may be determined once for all and recorded or engraved upon it, and if that weight be a simple number of weight units, such as 10 Gm or 5 Gm, the division of the weight of the other liquid by the weight of the same volume of water is extremely simple.

316. When solids of different densities are mixed with each other and it is desired to separate the heavier from the lighter this separation may be effected by adding a liquid which is lighter than the heavier solids and heavier than the lighter solids.

317. A *Pycnometer* is a bottle constructed so that it can be conveniently used for the determination of the densities of liquids. Pycnometers are often called "specific gravity bottles."



Fig. 105. Plain pycnometer with counterpoise.

Several such bottles are figured here.

The bottle should be made to hold exactly 25 or 50 or 100 Gm of pure water at 15° C.; but many pycnometers are adjusted to the temperature of 15.°5 instead of 15°. The best pycnometers are cylindrical rather than round, of light weight (thin glass), provided with well ground stoppers with vertical capillary tubes bored through their centers. Or the stopper of the pycnometer may be a thermometer, while a second longer neck is provided with a cap or a perforated ground stopper. The instrument may be accompanied by a counterpoise.

When the pycnometer is to be used it is filled to the brim with the liquid to be tested, and the stopper or stoppers then carefully but firmly inserted, after which the outside of the instrument is washed with water or alcohol if necessary and wiped dry with blotting paper, and then weighed with its contents. The tare or weight of the bottle must be deducted from the total weight if a counterpoise is not used. The temperature at which the operation is performed is to be observed and the result noted and used accordingly. This is rendered easy when a thermometer is fitted into the bottle as a stopper to it; otherwise the temperature of

the liquid must be taken by a thermometer immersed together with the filled pycnometer in a bath and allowed to remain there until of the desired temperature throughout its contents.

318. Adjustment of the temperature. It is very difficult to impart to any liquid the precise temperature at which its density is to be found, and to maintain that temperature until the observations are completed. A bath can of course be used in which water is cooled with the aid of ice until a thermometer inserted in the liquid indicates the temperature of a little below 15° C., after which the unmelted portion of the ice may be removed; but the temperature of the water must soon rise again above 15° if the temperature of the room is much above 15. It is, therefore, preferable to take the densities of liquids at any observed temperature near 15° C., and to make corrections afterwards for the contraction caused by a lower temperature or the expansion caused by a higher temperature.

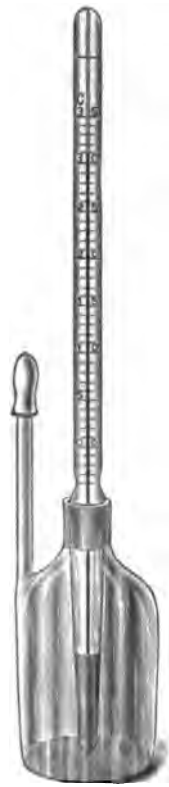


Fig. 106. Pycnometer with thermometer.

When pycnometers with thermometers are used the bottle may be filled with a liquid having a temperature a little below 15°, and may then be held in the hand, with filter paper between the hand and the bottle, until warmed to 15°. Hydrometer jars containing liquids may be warmed in the same manner; but the hydrometer itself must then also be warmed at the same time so that the process is here much slower.

319. Tables of coefficients which must be used to make the required corrections for deviations from the standard temperature will be found in special works on weights, measures and specific gravity, and in other books.

320. *Graduated flasks* are quite convenient and sufficiently accurate for ordinary determinations of density. They should be thin flasks, of globe-shaped or pear-shaped bodies with long slender necks, and so constructed as to hold 50, or 100, or 200, or

500, or 1,000 Grams of distilled water at 15° C. when filled to the graduation mark etched upon and around the neck.

Such flasks are often marked with an etched inscription stating their capacity in Cc. at 15.°; but they are, of course, graduated by weight, the weight does not change with variations of tem-



Fig. 107. Dr. Squibb's pycnometers.

perature, a cubic-centimeter is a Cc. at any temperature, and the cubical expansion or contraction of the glass flask itself is not considered.

To find approximately the density of any liquid, weigh it in the graduated flask, and divide by the weight of the same volume of water.

321. Hydrometers. A floating body displaces its own weight of the liquid in which it floats—no more, and no less. Hence it sinks deeper in lighter liquids, and to a less depth in heavier liquids. Hydrometers are cylindrical floats so constructed that the center of gravity is at one end, with an expanded part of the tube just above the loaded bulb which fixes the center of gravity, the object of the expansion being to keep the instrument in an upright position when floating in a liquid. Several forms of hydrometers are represented by the illustrations.

Hydrometers, or areometers, are much used in laboratories. The most useful and common forms, only, will be referred to here.

A hydrometer which is heavier than its own volume of the liquid in which it is placed would, of course, sink below the surface of that liquid if its depth is sufficient; and a hydrometer not sufficiently heavy, or not properly constructed, would not sink down far enough to assume a vertical position. But a hydrometer so constructed as to sink down to about the middle in water might be used for liquids heavier than water as well as for liquids lighter than water. In order, however, to render the hydrometer convenient as well as accurate, each instrument is made to indicate only a limited range of densities, and the tube above the expanded part which helps to constitute the instrument an upright float is made sufficiently long to render the divisions on the graduated scale large and distinct enough to be easily read but not so long as to make the hydrometer unwieldy.

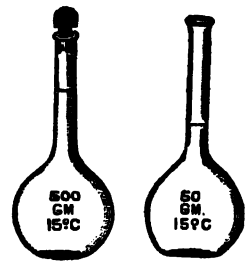


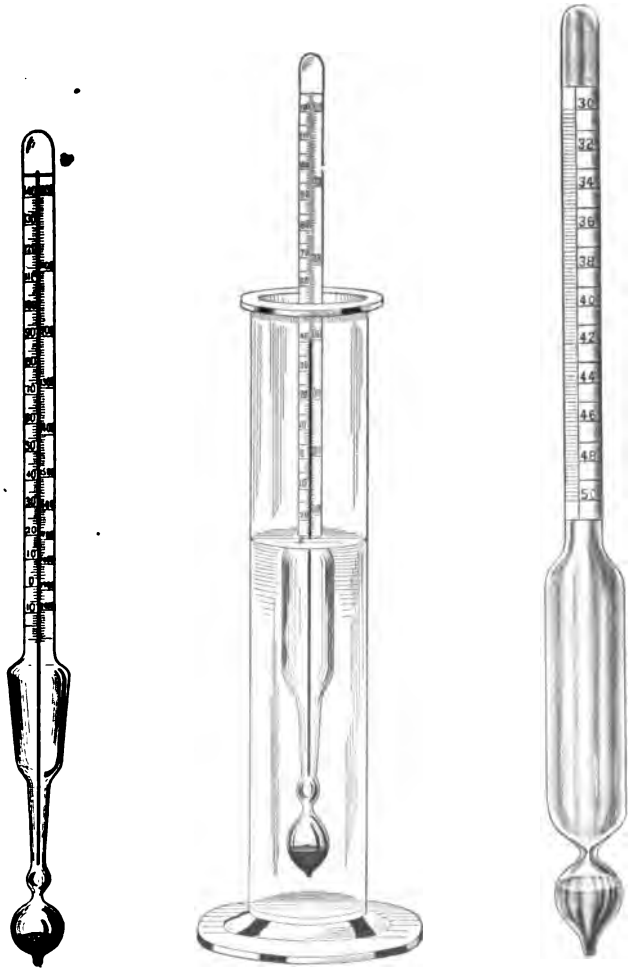
Fig. 108. Graduated flasks.

322. The two most common hydrometers are: one for densities ranging from 1.000 up to 1.300; and the other for densities ranging from 1.000 down to 0.700. The first-mentioned is far more commonly used than any other, because liquids heavier than water but not heavier than 1.300 sp. gr. are the most common liquids to be tested—salt solutions, etc. A hydrometer for liquids between 1.300 and 1.600 sp. gr. is also made, and another for liquids ranging from 1.600 to 2.000; but these are rarely used.

The four hydrometers just described are called "direct specific gravity hydrometers" because the scales within or upon their slender tubes are graduated to indicate actual densities.

323. Very good hydrometers are rarely accurate to within

0.002 sp. gr., and many instruments fail to indicate less differences than 0.010. Hydrometers are therefore much less reliable than the Mohr-Westphal balance, the pycnometer, or the graduated narrow-necked specific gravity flask (Par. 320); but their con-



Figs. 109 and 110. Hydrometers.

venience is so great that hydrometers are largely employed whenever approximately correct results are sufficient. Hydrometers indicating differences of 0.005 may be regarded as extremely

satisfactory for all ordinary laboratory operations in the manufacture of chemical products.

The spaces between the graduation marks on the hydrometer scales are always larger at the top than lower down.

324. The *hydrometer jar* (Fig. 110) must be sufficiently roomy to allow the hydrometer to float freely without coming in contact with the sides, and tall enough to leave the upper end of the hydrometer protruding above the top of the jar when the instrument is immersed in the lightest liquid, the density of which is included within the range of the scale.



Fig. 111. Rousseau's densimeter.

325. If it is desired to take observations of density at 15° the hydrometer and jar must both be cooled with cold water before they are used.

The best hydrometers include *thermometers* within their tubes so that the temperature can be observed concurrently with reading off the density.

326. The *meniscus* renders the reading difficult, but care and experience will enable the operator to obtain satisfactory results; the density mark at the middle of the depth of the meniscus indicates the correct specific gravity.

327. Large hydrometers demand the use of much liquid, and smaller hydrometers (each made for a more limited range of densities) are accordingly often preferred, or even necessary.

328. *Twaddell's hydrometers* are comparatively small, as the graduated scales of these hydrometers occupy a space only about 75 millimeters long. They are made in sets of six, and as the whole range of densities divided up between these six instruments extends only from 0 to 174 *degrees* or from the sp. gr. 1 to 1.870 the degree spaces are large enough to be easily read. The zero on Twaddell's scale corresponds to the density of water at 15.°6 C. (60° F.), and each degree above the 0 represents an increase of 0.005 of the density of water, so that 2° Twaddell is equivalent to the sp. gr. 1.01, and 10° Tw. to sp. gr. 1.05.

It will be observed that Twaddell's hydrometers are intended only for liquids heavier than water.

On account of their small size the Twaddell hydrometers can be used in the customary graduated cylinders of 100 Cc. capacity, so that no special hydrometer jar is necessary.

To convert Twaddell degrees into the corresponding specific gravity *multiply the number of degrees by* 0.005 *and add* 1. *to the product.*



Fig. 112. Twaddell's hydrometer.

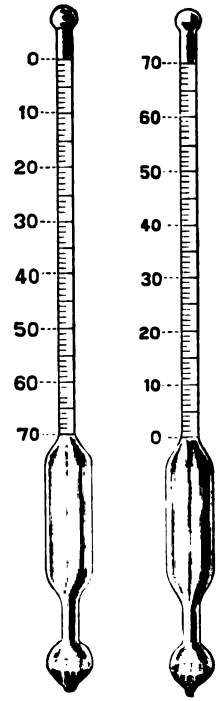


Fig. 113. Baumé's hydrometers.

329. Baume's hydrometers were once very generally used; but are falling into disuse on account of their very unscientific scales of degrees, and their uncertain standards.

The Baumé hydrometer for *heavy* liquids is assumed to descend to 0 in water at 12°.5 C., and to descend to the point marked 15° in a solution made of 15 parts of sodium chloride in 85 parts of water.

The Baumé hydrometer for *light* liquids is assumed to sink to 0 in a 10 per cent solution of sodium chloride at 12.°5 C. and to 10° in pure water.

The Baumé hydrometer should be altogether discarded, but as "degrees Baumé" are referred to in many valuable old works the following rules for converting Baumé degrees to sp. gr. and *vice versa* are here given. These rules refer to Baumé scales used in the United States and hold good at the temperature of 15.°6 C. (60° F.).

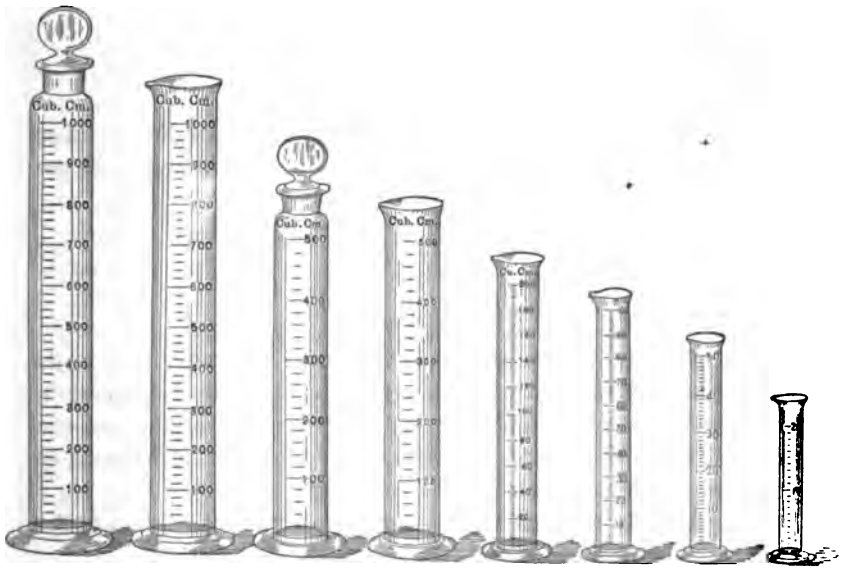


Fig. 114. Graduated cylinders.

For liquids heavier than water.

I. To convert Baumé degrees into specific weight, divide 145 by the remainder obtained by deducting the number of Baumé degrees from 145:

$$\frac{145}{145-B^\circ} = \text{sp. w.}$$

II. To convert specific weight into the corresponding degrees

Baumé, subtract from 145 the quotient obtained by dividing 145 by the specific weight:

$$145 - \frac{145}{\text{sp. w.}} = \text{B°}.$$

For liquids heavier than water.

III. To convert Baumé degrees into specific weight, divide 140 by the sum of 130 plus the number of degrees:

$$\frac{140}{130+\text{B°}} = \text{sp. w.}$$

IV. To convert specific weight into the corresponding number of degrees Baumé, divide 140 by the specific weight and from the quotient subtract 130:

$$\frac{140}{\text{sp.w.}} - 130 = \text{B°}.$$

330. The knowledge of the density of a substance aids in its identification, and in the determination of its purity and strength, and enables us to compute the weight from the volume and the volume from the weight. It is also a guide in the concentration of solutions by evaporation for various purposes.

331. Specific volume.* The specific volume of a liquid is the reciprocal of its specific weight.

Its utility in laboratory work is great.

To convert any number of grams into the corresponding number of milliliters, or any number of kilograms into the corresponding number of liters, *multiply by the specific volume.*

*The term specific volume and its employment were proposed by the author of this book in 1883, in a paper read before the American Pharmaceutical Association. See Proceedings of that Association for the year named.

CHAPTER XVIII.

RULES FOR MAKING SOLUTIONS AND MIXTURES OF ANY GIVEN STRENGTH, AND FOR DILUTING, FORTIFYING AND MIXING.

332. To make solutions and mixtures of any given percentage strength all the quantities and values employed should refer to weight.

Accurate results are obtained when all the materials are measured by weight, because mass is not affected by contraction or expansion of volume.

But when the quantities of the materials or ingredients are measured by volume instead of by weight, or when the quantity of one of two ingredients is measured by weight and that of the other by volume, the exact strength of the mixture or solution can not be directly or easily computed or expressed.

Even if the mixture or solution is composed of two liquids, their proportions should be stated and computed by weight and not by volume, because the volume of the resulting mixture or solution is generally less than the sum of the original volumes of the two component liquids, so that the proportion which the volume of either of these two liquids bears to the final volume of the product can not be known from the proportions employed. A mixture of alcohol and water, one hundred volumes of which contain fifty volumes of absolute alcohol, contains an amount of water which separately must measure more than fifty volumes. A statement of percentage strength or composition referring to volume proportions is accordingly unreliable and deceptive.

Nevertheless it is so convenient for many pharmaceutical purposes to prepare solutions and liquid mixtures volumetrically, or to measure the solids by weight and the liquids by measure in making preparations composed of both, that it is a common practice, and the results are *in most cases* sufficiently nearly accurate for those purposes.

When absolute exactness is necessary, however, all quantities and values employed in working-formulas, in actual work, and in statements of strength referring to liquids must be expressed in

terms of weight exclusively. Gravimetric methods, formulas and expressions are always practicable and exact, and for nearly all purposes in chemical laboratory work they are preferable in point of convenience as well as scientific accuracy.

333. The following—

Rule for computing quantities and proportions in making mixtures and solutions will be found applicable in most cases:

Having a known quantity (a) of one ingredient of known value (A), and desiring to find the quantity (x) of the second ingredient of known value (B) which will be required to produce a mixture of any intermediate value (c)—*Multiply the known quantity of the first ingredient* (a) *by the difference between its value* (A) *and the desired value of the mixture* (c); *divide the product by the difference between the value of the second ingredient* (B) *and the desired value of the mixture* (c). *The quotient is the quantity desired of the second ingredient* (x).

For the purposes of the foregoing rule the term "ingredient" applies to each of the two components of any mixture or solution. Thus the two ingredients of any solution are the solvent and the dissolved substance; the ingredients of any dilute alcohol are the alcohol and water; and the components of any mixture of two solutions of different strength, or of a mixture of a stronger and a weaker alcohol, or of a mixture of a stronger and a weaker opium, are also designated as "ingredients."

The "value" of each ingredient as well as the value of the mixture must be expressed in per cent by weight.

If the "ingredients" are water and potassium bromide, then the value of the water is 0 and the value of the bromide 100; in a mixture of absolute alcohol and water, the alcohol has a value of 100 and water 0. If opium is one ingredient and milk sugar the other, the value of the opium may be expressed as 100 and that of the mik sugar as 0 *if the value of the mixture is to be expressed in terms per cent of opium;* but if the value of the opium be expressed in per cent of morphine, then the strength of the milk sugar and of the mixture must also be expressed in the same terms. Thus if an opium containing 16 per cent of morphine is

to be diluted with milk sugar to 14 per cent, then the value of the opium in this case must be 16 and that of the milk sugar 0.

If a mixture is to be made of alcohol and glycerin, to contain a definite per cent of alcohol by weight, then the value of the alcohol for the purposes of the application of our rule will be 100 and that of the glycerin 0; but if the value of the mixture is to be expressed according to the per cent of glycerin it contains, then the value of the alcohol will be expressed by 0 and that of the glycerin by 100.

If a mixture is to be made of any two ingredients of unequal value, the rule is still applicable. It thus applies to the dilution or fortification of all solutions and mixtures in which two ingredients, one weaker and one stronger, are employed as the ingredients or components.

The term "mixture" includes, for the purposes of this rule, all solutions.

If we designate as (d) the difference between the value of the first ingredient (A) and the value sought (c), and as (D) the difference between the value of the second ingredient (B) and the desired value of the mixture (c), the rule may then be reduced to the following algebraic formula—

$$\frac{a \times d}{c \times D} = x.$$

It makes no difference which of the two ingredients is designated as "first" or "second."

Examples.

1. How shall a 30 per cent solution be made of potassium hydroxide and water?

The value of the first ingredient, KOH, is 100, and the difference between 100 and 30 is 70.

The value of the second ingredient, the water, is 0, and the difference between 0 and 30 is 30.

Designating the quantity of potassium hydroxide as 1, the application of the rule would give—

$$\frac{1 \times 70}{\cdot 30} = 2.333 = \text{the number of weight units of water to be}$$

added to each weight unit of potassium hydroxide.

2. How much water must be added to 30 Gm of potassium hydroxide to make a 30 per cent solution of it?

$$\frac{(100-30)\times 30}{30-0}=70.$$

Answer: 70 Gm of water.

3. How much zinc sulphate must be added to 200 Gm of water to produce a solution of 4 per cent strength?

The water may be called the first ingredient and has a value of 0. The zinc sulphate is the second ingredient and its value is 100. Then—

$$\frac{200\times(4-0)}{100-4}=8.333 \text{ Gm.}$$

Answer: 8.333 Gm of zinc sulphate.

4. How much solution of 3 per cent strength can be made out of 400 Gm of a solution of 18 per cent strength?

Let us find how much water must be added.

$$\frac{400\times(18-3)}{3}=2000 \text{ Gm.}$$

Answer: 2000 Gm of water must be added to the 400 Gm of 18 per cent solution, producing, therefore, 2400 Gm of 3 per cent solution.

5. How much solution of 50 per cent strength must be added to 333.33 Gm of water to produce a solution of 30 per cent strength?

$$\frac{333.33\times(30-0)}{50-30}=500 \text{ Gm.}$$

Answer: 500 Gm of 50 per cent solution must be added to 333.33 Gm of water to produce 833.33 Gm of solution of 30 per cent strength.

6. How much zinc sulphate must be added to 600 Gm of a 20 per cent solution of that salt to increase the strength of the solution to 25 per cent?

$$\frac{600\times(25-20)}{100-25}=40 \text{ Gm.}$$

Answer: 40 Gm of zinc sulphate added to 600 Gm of a 20 per cent solution of zinc sulphate will make 640 Gm of a solution of 25 per cent strength.

7. How much of a borax solution of 5 per cent strength must be added to 800 Gm of a borax solution of 2 per cent strength to produce a solution of 3 per cent strength?

$$\frac{800\times(3-2)}{5-3}\ 400 \text{ Gm.}$$

Answer: 400 Gm of borax solution of 5 per cent strength added to 800 Gm of a solution of 2 per cent strength will produce 1200 Gm of borax solution of 3 per cent strength.

8. How much alcohol of 91 per cent must be added to 3000 Gm of alcohol of 41 per cent strength to produce a mixture containing 75 per cent of alcohol?

$$\frac{3000\times(75-41)}{91-75}=6375 \text{ Gm.}$$

Answer: 6375 Gm of alcohol of 91 per cent strength mixed with with 3000 Gm of alcohol of 41 per cent will make 9375 Gm of an alcohol of 75 per cent.

9. How much alcohol of 41 per cent strength must be added to 6375 Gm of alcohol of 91 per cent strength to produce an alcohol of 75 per cent?

$$\frac{6375\times(91-75)}{75-41}=3000 \text{ Gm.}$$

10. How much glycerin must be added to 864 Gm of alcohol to produce a mixture containing 10 per cent of glycerin?

$$\frac{864\times(10-0)}{100-10}=96 \text{ Gm.}$$

Answer: 96 Gm of glycerin.

11. How much glycerin must be added to 864 Gm of alcohol to produce a mixture containing 10 per cent of alcohol?

$$\frac{864\times(100-10)}{10-0}=7776 \text{ Gm.}$$

Answer: 7776 Gm of glycerin added to 864 Gm of alcohol will produce a mixture containing 10 per cent of alcohol.

12. How much opium containing 10 per cent of morphine must be added to 420 Gm of opium containing 18 per cent of morphine in order to make a mixture containing 13 per cent of morphine?

$$\frac{420\times(18-13)}{13-10}=700 \text{ Gm.}$$

Answer: 700 of opium of 10 per cent strength and 420 Gm of opium of 18 per cent strength when mixed will make 1120 Gm of opium of 13 per cent morphine strength.

334. To find the relative proportions required of salt and water to make 100 Gm of any salt-solution of any given strength:

Rule. *Subtract the desired per cent from* 100; the *remainder will show the number of Gm of water required. The number expressing the desired percentage strength of the solution will be the number of Gm of salt required.*

Ex. To make 100 grains of a 4 per cent solution of cocaine hydrochlorate use 4 grains of cocaine hydrochlorate and 96 grains of water.

335. To find the quantity of salt (a) which must be added to any given quantity of water (b) to produce a solution of any given per cent strength (c), and the quantity of solution obtained (d):

Rule. *Multiply the grams of water* (b) *by the desired per cent of the solution* (c) *to be made, and divide the product by the remainder found by subtracting the number expressing that per cent from* 100; *the quotient will be the number of grams of*

salt (a) which must be added to the grams of water (b). The sum of the number of grams of salt and water is, of course, the weight of the solution (d).

Formula—

$$\frac{b \times c}{100 - c} = a; \text{ and } a + b = d.$$

Explanation—

$(100 - c) : b = c : a$; and $a + b = d$.

Ex. To find the number of Gm of mercuric chloride required to be added to ten kilograms of water to make a 0.1 per cent solution, multiply 10,000 by 0.1, and divide the product (100,000) by 100—0.1 (or 99.9). The quotient is 100.1 Gm, which is the quantity of mercuric chloride required.

336. To find the kilos of water (*b*) required for any given number of kilos of salt (*a*) to make a solution of any given percentage (*c*) strength, and the kilos of solution produced (*d*):

Rule. *Multiply the kilos of salt (a) by 100, and divide the product by the desired percentage strength (c) of the solution to be made. The quotient is the number of kilos of solution (d) which will be produced by the salt; and the difference between the kilos of salt (a) and the kilos of solution formed (d) will be the kilos of water required (b).*

Formula—

$$\frac{a \times 100}{c} = d; \text{ and } d - a = b.$$

Explanation—

$c : a = 100 : d$; and $c \times d = a \times 100$.

Ex. In making an 8 per cent solution out of 24 kilograms of sodium carbonate, how much solution will be obtained and how much water must be used?

$$\frac{24\times 100}{8}=300;\ \text{and}\ 300-24=276.$$

Answer: 300 kilograms of 8 per cent solution will be produced out of 24 kilograms of sodium carbonate and 276 kilograms of water.

337. To find the quantity of weaker solution of any given strength that can be made out of any given quantity of a stronger solution, and thus also the quantity of diluent required:

Rule. *Multiply the grams of the stronger solution* (*a*) *by its percentage strength* (*b*), *and divide the product by the desired lower percentage strength* (*c*); *the quotient is the grams of weaker* solution (*d*) produced by the stronger solution (*a*). The difference between *d* and *a* is the number of grams of diluent required (*e*).

Formula—

$$\frac{a\times b}{c}=d;\ \text{and}\ d-a=e.$$

Explanation—

$$c:b=a:d;\ \text{and}\ c\times d=a\times b.$$

Ex. If 200 Gm of a solution of 50 per cent is to be diluted to 30 per cent—

$$\frac{200\times 50}{30}=333.33;\ \text{and}\ 333.33-200=133.33.$$

Answer: 333.33 Gm of the 30 per cent solution can be made from 200 Gm of 50% solution, so that the quantity of water to be added is 133.33 Gm.

338. To find the quantity of stronger solution required to make any given quantity of weaker solution, and thus also the quantity of diluent required:

Rule. *Multiply the required quantity of the weaker solution (a) by its desired per cent strength (b), and divide the product by the percentage strength (c) of the stronger solution; the quotient is the quantity required (d) of the stronger solution. The quantity of diluent required (e) is the difference between a and d.*

Formula—

$$\frac{a \times b}{c} = d; \text{ and } d - a = e.$$

Explanation—

$c : b = a : f$; and $c \times d = b \times c$.

Ex. How much 25 per cent solution will be required to make 600 Gm of a 20 per cent solution? And how much water?

$$\frac{600 \times 20}{25} = 480; \text{ and } 600 - 480 = 120.$$

Answer: 480 Gm of 25% solution and 120 Gm of water will make 600 Gm of 20% solution.

339. To find the kilos of water (x) required to be added to each kilo of a solution of any given percentage strength to dilute it to any less percentage desired:

Rule. *Divide the percentage strength (a) of the stronger solution by the percentage desired (b), and subtract 1 from the quotient.*

Formula—

$$\frac{a}{b} - 1 = x.$$

Ex. A 90% solution is to be diluted to 60%. How many kilos of water must be added to each kilo of the 90% solution?

$\frac{90}{60}$ = 1.50; and 1.50 — 1 = 0.50.

Answer: 0.50 kilo of water must be added to each kilo of the 90 per cent solution.

340. To find the absolute quantities of salt (a) and water (b) required to make any given quantity of solution (c) of any given strength (d):

Rule. *Multiply the grams of solution required (c) by the desired percentage strength (d), and divide the product by* 100. *The quotient is the grams of salt required (a). The grams of water (b) required is the difference between the grams of salt (a) and the grams of solution (c).*

Formula—

$$\frac{c \times d}{100} = a; \text{ and } c - a = b.$$

Explanation—

$100 : d = c : a$; and $100 \times a = c \times d$.

Ex. How much sodium hydroxide is required to make 500 Gm of a 6% solution? And how much water?

$$\frac{500 \times 6}{100} = 30; \text{ and } 500 - 30 = 470.$$

Answer: 30 Gm of sodium hydroxide and 470 Gm of water.

341. To find the percentage strength of any salt-solution from the grams of salt contained in a given number of grams of solution:

Rule. *Multiply the grams of the salt (a) by* 100 *and divide by the grams of the solution (b); the quotient is the percentage (c).*

Formula—

$$\frac{a \times 100}{b} = c.$$

Explanation—

$b : 100 = a : c$; and $b \times c = 100 \times a$.

Ex. If 2.25 Gm of absolute acetic acid be contained in 50 Gm of a water solution of it, the percentage strength of the solution must be 4.50%, for—

$$\frac{2.25 \times 100}{50} = 4.50.$$

342. To find the percentage strength (a) of a solution from the weight of the solvent (b) and of the solution (c):

Rule. *Subtract the weight of the solvent (b) from the weight of the solution (c); multiply the remainder by* 100, *and divide the product by the weight of the solution (c). The quotient is the percentage (a).*

Formula—

$$\frac{(c-b) \times 100}{c} = a.$$

Explanation—

$(c-b) : c = a : 100$; and $c \times a = (c-b) \times 100$.

Ex. If 1000 Gm of water be saturated with a salt and the total weight of the solution formed be found to be 1100 Gm, what is the percentage strength of the solution?

$$\frac{(1100-1000) \times 100}{1100} = 9.0909.$$

Answer: 9.09 per cent.

334. *Alligation* is frequently a convenient method of finding the proportions required of two or more ingredients of different values required to produce a mixture of any value intermediate between the highest and lowest.

To find the proportions required of any two ingredients of different values required to produce a mixture of any desired intermediate value.

Rule. *The number of parts required of the weaker ingredient* (*a*) *is the difference between the value* (*percentage strength*) *of the stronger ingredient* (*b*) *and the desired value of the mixture* (*c*).

The number of parts required of the stronger ingredient (*d*) *is the difference between the desired value* (*percentage strength*) *of the mixture* (*c*) *and the value of the weaker ingredient* (*e*).

$a=b-c$ and $d=c-e$.

Ex. To make a mixture of 7 per cent strength out of one of 3 per cent and another of 12 per cent, use $12-7=5$ parts of the 3 per cent ingredient and $7-3=4$ parts of the 12 per cent ingredient.

CHAPTER XIX.

FURNITURE AND APPARATUS.

335. The outfit of apparatus for a pharmaceutical laboratory in which chemical preparations are made must vary to so great an extent according to the quantity and variety of the products, and according to other circumstances, that a definite description of such a laboratory is scarcely practicable. A few general suggestions may, however, be found useful to pharmacists as well as students who desire to undertake to make chemical preparations.

Certain laboratory furniture, fixtures and apparatus are, of course, always necessary.

These include wall fixtures, such as shelving, cupboards, a case of drawers, good water-supply, one or more sinks, good ventilation, one or more effective flues with which to connect a drying closet and a hood or fume chamber, work tables, heating apparatus, a good still for making distilled water, a press, and a sufficient outfit of pots and pans, jars, bottles, strainer frames and stands, dishes, funnels, filter stands, glassware, etc.

FURNITURE AND FIXTURES.

336. If the products made include considerable quantities of precipitates and crystallized salts requiring drying, a **drying closet** is of great advantage. In large laboratories two or more drying closets kept at different temperatures are more serviceable than one large one; or a special drying room and one or two closets are used. Small drying closets may be heated by means of steam coils, hot water coils, or hot air, according to circumstances. Where steam or hot water can not be had the drying closet may be heated by means of a suitable gas burner or gas stove, or by a gasoline or oil burner, placed at the bottom. The closet must, of course, be provided with openings to admit air below the heating appliance used, and also at the top so that a current is established. The top of the closet should be connected with an effective flue, if possible; but if this can not be done some other means of establishing a draft should be adopted. The current of air

passing through the drying closet need not (and, for most purposes, *should* not) be strong. Racks for shelves or frames are so arranged that the circulation of air is not impeded. For some purposes it is admissible to use shelves made of galvanized iron wire grating. Strips of plate glass, wood, or tinned iron are often employed instead of shelves.

The moist products to be dried in the drying closet are spread out upon filter paper or muslin placed on the shelves or strips. It is important to regulate the temperature well so that substances liable to injury from too high heat may not be spoiled.

337. A tight **fume chamber** or hood connected with a flue having a good draft is essential if operations are to be performed resulting in the evolution of irritating or poisonous gases, such as nitrous vapors, chlorine, sulphur dioxide, etc. The fume chamber should have a soapstone bottom, and at least two sides of it should consist of movable sash with double thick window glass.

338. The **work tables** must be strong and heavy. They should be about 0.9 meter high and not over 0.6 meter wide if placed lengthwise against the wall, but about 1 meter wide if out from the wall.

One of the tables should have a tin covered top inclined toward and adjoining the sink, and this table should be exclusively for apparatus just cleaned and left to be drained.

Another table adjoining the sink should be covered with sheet lead and should have a raised edge (also covered) all around it so that operations in which corrosive or inflammable liquids are used may be performed more safely.

Soapstone tables are most desirable for operations requiring the continuous use of powerful burners.

Other work tables or counters should have wooden tops.

339. If practicable there should be in any large laboratory a specially planned place for stills, pans, strainer stands and the press—a place where the **floor is covered with asphalt** and inclined toward a drain so that the water which is frequently freely used in connection with that kind of apparatus will readily run off. If an asphalt floor is impracticable a **tin-covered,** low, inclined **platform** with raised edges is useful. A plentiful and convenient supply of water with taps or faucets wherever required is very important here.

The waste-pipes from steam kettles, stills, etc., are to be connected with an ample outlet.

BALANCES, ETC.

340. A special room or place for the *balances* is necessary in all large laboratories. In a pharmacy where the laboratory consists of a work-room adjoining the officine, all *weighing and measuring* may conveniently be done with the same instruments that are used for other work. In large manufacturing laboratories the materials required by the laborants are supplied from a general store-room upon special orders, and the analytical work is done in another room specially equipped for that purpose.

A well-furnished pharmaceutical establishment, even if very little manufacturing is done in it, should have several balances. Balances are now made in the United States which are in no wise surpassed by any foreign made instruments of the same kind.

The most suitable balances for a well ordered pharmaceutical establishment are:

1. An **analytical balance** upon which quantities ranging from 1 milligram to 50 grams may be weighed. When *not loaded* this balance should be sensitive enough to respond to 1 milligram by a 2 millimeters' deviation of the point of the needle from the center of the dial; when this balance is *loaded* to the extent of 50 grams the needle point should deviate 2 millimeters from the center of the dial on the impulse of 5 milligrams.

2. The ordinary **prescription balance** should be used to carry loads of from 5 milligrams to 5 grams. The sensitiveness of this balance should be such that when the pans are *not loaded* the needle should deviate 2 millimeters from the center of the dial under the impulse of 5 milligrams; when the pans are *loaded* with 50 Gm each the point of the needle should deviate 2 mm from the center on the impulse of 20 milligrams.

3. The **gram balance** should be designed for loads of from 1 to 250 Gm. Its sensitiveness should be such that when the pans are *not loaded* the impulse of 20 milligrams placed on one pan should be sufficient to cause the needle to deviate 2 millimeters from the center of the dial; and when the pans each carry a load of 250 Gm, the addition of 50 milligrams to the load on one pan

should cause the needle to deviate the same distance from the center.

4. The **kilogram balance** should be constructed to carry loads of from 5 to 2000 Gm, and its sensitiveness should be such that the needle point deviates from the center by 2 millimeters on the impulse of 50 milligrams when the pans are *not loaded* and on the impulse of 200 milligrams when they are loaded with 2000 Gm each.

One or two *larger balances* may also be necessary.

A *Mohr-Westphal balance* is of great value.

All balances should be placed on solid tables where they will be safe against jarring and injury, and must be kept well protected against dust. They should be kept scrupulously clean and dry, and no chemicals should be permitted to come in contact with them. They should above all be protected against corrosive substances. When not in actual use they must be at perfect rest, with the knife-edges relieved from friction.

The *weights* should also be kept clean and accurate.

341. Graduated glass measures, commonly called "graduates," are necessary in every pharmaceutical establishment. They should be graduated in accordance with but one system; those graduated according to the old system of fluid measure on one side and in cubic-centimeters on the other side, should never be used for any purpose because such double graduation leads to frequent errors.

The metric system having now been adopted in every pharmacopœia, the world over, pharmacists should do all in their power to shorten the time required to complete the change from the old to the new, not only because resistance to the inevitable is useless but because they will certainly find the new system far more convenient.

Graduated cylinders, flasks, pipettes, burettes, an accurate pycnometer, and a set of reliable hydrometers are required for the analytical work inseparable from the work of producing and dispensing chemical preparations. (See Chapter XV.)

HEATING APPARATUS.

342. Much can be done with the stoves, furnaces and burners constructed for use with gas as the fuel. Illustrated descriptive

catalogues of such heating apparatus are freely furnished by dealers and manufacturers so that any one may learn more from them than can be presented in any text-book. Among the most efficient **gas burners** are the "Fletcher low temperature burner,"

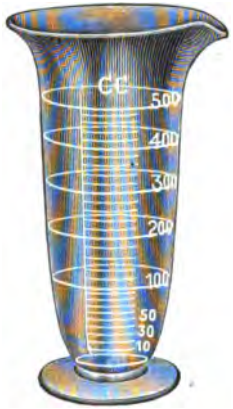


Fig. 115. Graduated glass measure.



Fig. 116. Fletcher burner ("low temperature burner").

which affords a wide range of temperatures from gentle heat up to red heat; it is so constructed that it can be attached to a pipe from a bellows when a very high heat is to be produced, and it

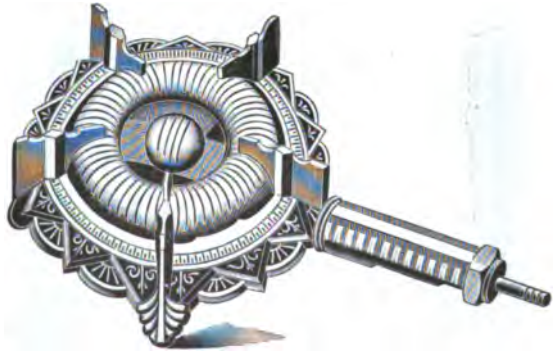


Fig. 117. Fletcher radical burner.

is so effective that the designation "low temperature burner" is an unfortunate one since it emphasizes one of its merits—that the heat can be thoroughly controlled and kept very low if desired—

at the expense of the equally valuable feature which enables the operator to apply an exceptionally high heat.

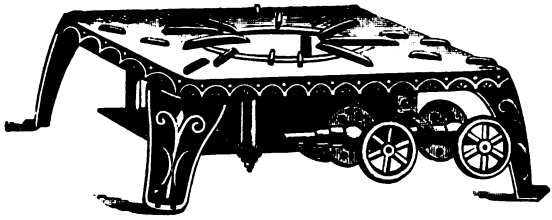


Fig. 118. The Jewel gas heater.



Fig. 119. Foot blower.



Fig. 120. Iron retort for the production gases in "the dry way" and for other processes requiring very high heat.



Fig. 121. Triple Bunsen burner.



Fig. 122. Bunsen burner with cone.

The Fletcher "radial burner" (made by Buffalo Dental Mfg. Co.) and the "Jewel" gas stove (made by Geo. M. Clark & Co., of Chicago) are particularly serviceable for heating large vessels.

A foot-blower or bellows is frequently useful.

Bunsen burners are made in great variety, and two or more



Fig. 123. Seven-tubed Bunsen burner.



Fig. 124. Erlenmeyer's burner.

well-made ones are necessary in any pharmaceutical laboratory, however small.

Bunsen burners are so constructed that a mixture of gas and air in proper proportions is first produced and more complete and rapid combustion and a maximum temperature are secured by burning that mixture.

The supply of gas admitted through the tube of the Bunsen burner can be regulated by the gas stopcock, and the supply of air by means of the movable collar which is provided with two opposite circular openings acting as a valve which may be partially or entirely closed by turning the collar so as to place it in the required position opposite the openings in the lower outer tube of the burner, immediately below the top of the gas tip which it surrounds, as seen in the cut.

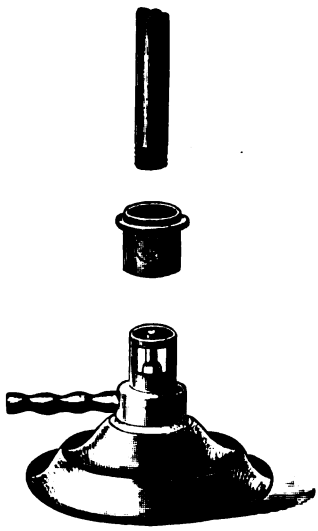


Fig. 125. Shows the construction of the Bunsen burner.

343. When sufficient air is supplied in proportion to the gas,

the flame is non-luminous or bluish, because the combustion is then complete; but when the supply of air is shut off the flame is luminous, yellow and smoky. When the current of gas is too low in proportion to the air admitted it frequently happens that the gas *ignites back,* the flame receding down into the tube to the pin hole orifice of the gas tip. The whole tube then at once becomes very hot so that the rubber tube attached to the burner may melt, the escaping gas then becoming ignited and a destructive fire may result. It is, therefore, dangerous to leave a Bunsen burner in operation with the gas turned low. Whenever it is found that the flame of the Bunsen burner has receded to the

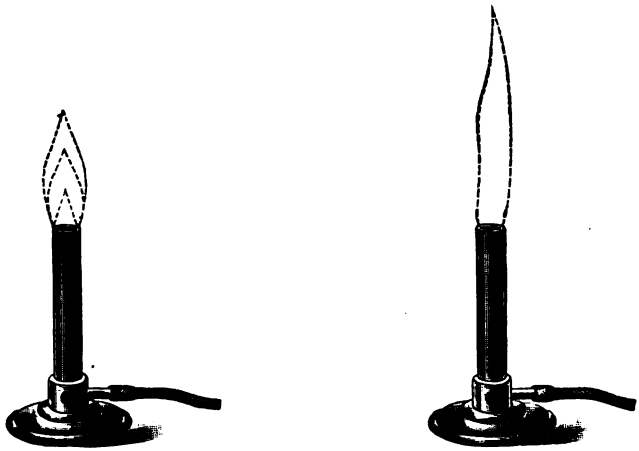


Fig. 126. Bunsen burner flames; the cuts contrast a proper flame with that resulting when the gas is "lighted back" into the tube.

base or "lighted back" the gas should be immediately shut off, and the burner allowed to cool off completely before it is again re-lighted; it may be cooled off quickly by letting cold water flow upon it.

344. In cases where it is necessary to allow a Bunsen burner to remain lighted a long time without constant attention, it should be placed on a *fire-proof table* and the gas should be supplied to it not by means of rubber tubing but through a piece of gas-pipe directly attached to the burner.

345. The introduction of a diaphragm of fine wire netting in the tube, just below the top, prevents the flame from "striking back," but it also reduces the intensity of the heat of the flame.

The mixture of gas and air passing through the wire netting is ignited above it. Gas burners constructed on this principle can be easily obtained.

Special burners are made for use with acetylene as the fuel, and acetylene burners produce a much higher temperature than gas burners.

346. The Roessler gas furnace is a very efficient apparatus for small crucible operations where extremely high temperatures are necessary. It can be used with either gas or acetylene.

Its construction is shown in the accompanying cut. It will

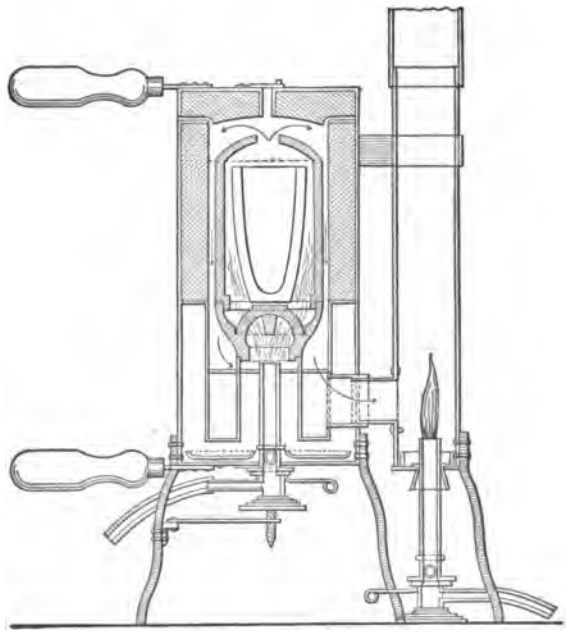


Fig. 127. Rœssler furnace.

heat a crucible about twelve centimeters high having a diameter of seven centimeters at the top. The length of the chimney may be extended to about two meters and to insure a strong draft through it a second burner is used at its foot.

347. When gas is not available *gasoline* burners may be advantageously used, and in many cases a good *coal* stove or a *charcoal* furnace may be found useful.

Spirit lamps are extremely valuable for small and brief opera-

tions where a perfectly smokeless flame and high heat are desired.

348. In the use of Bunsen burners spirit lamps and other burners which afford one solid vertical *flame,* the most intense heat is just below the middle of the flame.



Fig. 128. Barthel's spirit lamps.

349. When a vertical flame is used it is often necessary to distribute and moderate the heat by interposing *wire-gauze* or *asbestos-cloth* between the flame and the vessel heated. The gauze or cloth may sometimes incidentally serve as a support for flasks, beakers and other vessels to which the heat is applied. But wire gauze is soon "burnt out." Asbestos cloth is much to be preferred.

350. *Sand-baths* are frequently useful for supporting flasks, retorts and dishes which are to be strongly heated, serving to

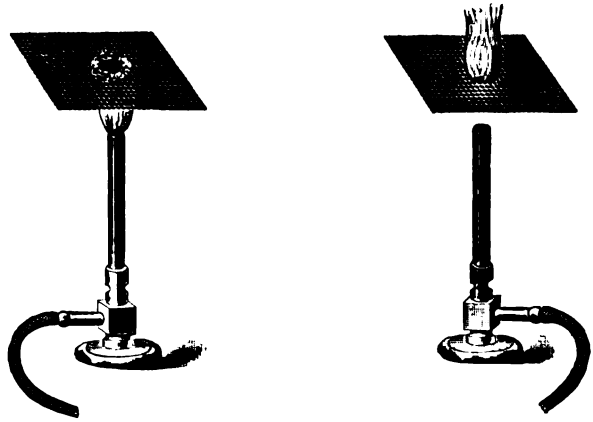


Fig. 129. Showing the effect of the interposition of wire cloth; the gas may be lighted either above or below the wire cloth.

distribute the heat. Shallow sand-bath dishes are used for the support of beakers and evaporating dishes and also flasks; deeper sand-bath dishes are necessary for heating retorts which must

sometimes be completely covered with the sand. Two forms of sand-bath dishes, made of the best "Russia iron" are shown in the cuts. A still deeper sand-bath is occasionally required. Iron pots are used for large sand-baths.

The *sand* used for the sand-bath should be clean, hard flint sand, not too fine but free from gravel.

The interposition of the sand-bath between the flame and the

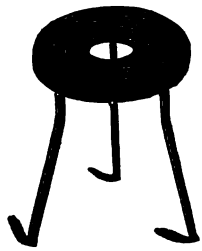


Fig. 130. Tripod with rings. For use with burners.



Fig. 131. Support for use with a Bunsen burner.

vessel distributes the heat very effectively; but it also causes considerable loss of heat and lowers its intensity.

351. Water-baths are round vessels of copper, or of heavy tinned iron, with either spherical or flat bottoms. Copper vessels are by far the best because, if made of moderately heavy sheet



Fig. 132. Shallow sand-bath dish of Russia iron.



Fig. 133. Deeper sand-bath dish of Russia iron.

copper and given reasonable care, they last for many years of constant use and because copper is an excellent conductor of heat.

Water-baths are indispensable in pharmaceutical laboratories. One or two sizes are required, if not more. For general use in comparatively small operations the best water-baths are those provided with sets of concentric rings to fit different sizes of dishes or flasks.

Water-baths without rings are used for special purposes.

Water-baths with *constant level* are very convenient, for the

ordinary water-bath requires watching and frequent replenishing with water to prevent their running dry or too low, which usually results in damage. The constant level water-bath is so constructed that the water vapor does not escape, but is condensed and runs back into the body of the vessel. The bath shown in Fig.



Fig. 134. Small round-bottomed copper water-bath.



Fig. 135. Small flat-bottomed water-bath.



Fig. 136. Water-bath with attachment to maintain a constant level of the water.

137 is, as seen, differently constructed. Various kinds may be found described in the catalogues of dealers in chemical apparatus.

The water-bath when in use should be kept from one-half to three-fourths filled with water, and the vessel placed upon it should not fit so tightly that there is no escape for the steam, for the pressure may then become so great as to throw the vessel

off the bath or to cause a sudden outburst of steam which might do injury.

The temperature afforded by the water-bath when well managed may be made to range all the way from the ordinary room temperature up to about 95° C.

352. *Salt solutions* (for from 100° to 160°), *glycerin* (up to 165°), and *paraffin* (up to 250°) are frequently employed in baths instead of water for the purpose of imparting heat. The temperatures afforded vary according to the boiling points. When temperatures below the respective boiling points of these several liquids are required it is necessary to use *thermometers* in order to be able to guard against a higher heat than that desired. Thermometers are especially necessary in water-baths when the temperature must not be permitted to exceed a given degree. At the same time the water-bath may then be placed a greater or less distance above the flame, and the flame or fire may be regulated as circumstances require.

353. *Hot water coils* may be advantageously used to heat sand or water in large sand-baths or water-baths employed in the practice laboratories of technical schools for the use of the classes. Such sand-baths and water-baths may be made to afford a reasonably uniform temperature anywhere between 30° and 70°.



Fig. 137. Instantaneous water heater.

354. *Hot air chambers*, heated with steam or hot water, and tightly closed, may be employed for maintaining nearly uniform temperatures for various laboratory purposes. They are usually made of plate glass in frames of wood, and the temperature of the air in them is observed by means of thermometers.

Hot air ovens and *hot water ovens* of copper are used for drying small quantities of various substances.

355. Steam heat is invaluable in *large* manufacturing labora-

tories. Even small steam boilers of very simple construction are of great utility in moderately equipped pharmaceutical laboratories. Where steam power is used, the steam also furnishes the heat for pans, stills, etc.



Fig. 138. Small drying oven in use.

The temperature imparted by steam-heating apparatus may be regulated so that it can be kept within narrow limits at any

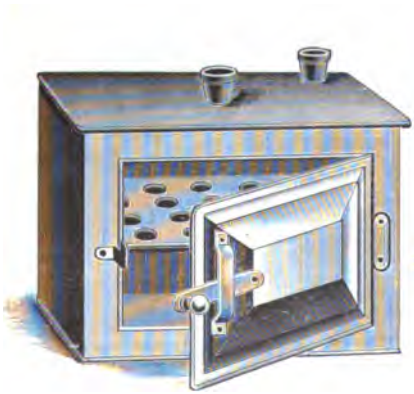


Fig. 139. Double-walled copper oven.

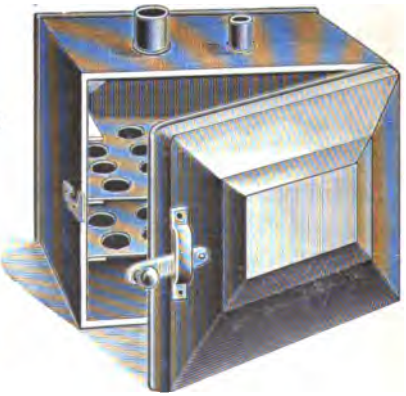


Fig. 140. Copper drying oven.

point within the extreme range of from a few degrees above the ordinary room temperatures up to 100°. The steam used may be under a pressure of from three or four pounds to the square

inch up to twenty-five pounds or more. For some special purposes, as in the fractional distillation of hydrocarbons on a large scale (in the manufacture of gasoline, benzin, lamp oil, etc.) the steam is conducted by means of iron pipes through a furnace fire to raise its temperature to a very high degree (even to a point at which it makes red hot the iron pipe through which it passes after leaving the furnace) before it is applied. Steam so treated is called *"superheated" or "dry steam."*

Steam heat is applied in three different ways: 1, by means of coils; 2, by means of jackets; and 3, by conducting it directly into the liquid to be heated.

A *coil* of steam pipe placed in the pan, kettle or still is very effective for heating the contents.

The *"steam jacket"* consists of an outer shell or second bottom riveted and soldered to the regular bottom of the pan, kettle or still, so as to make a steam-tight compartment between the two bottoms or shells. This "jacket" usually extends about one-third of the way up around the vessel, and leaves a space of about two inches between the shells at the center under it. The steam enters the jacket on one side near the center of the bottom through a horizontal pipe provided with a globe steam-valve. The condensed steam is let out through a vertical steam-pipe fitted to the outer shell at its lowest point, and this pipe also has a valve by which it can be opened or closed. The side pipe is connected by coupling with the regular steam supply pipe and the outlet pipe at the bottom is coupled to another connected with the "waste pipe."

A *jet* of steam issuing from the bottom of a single shell or pan through a pipe may also be used for heating vessels placed upon that shell and fitting it rather snugly, as a dish is placed over a water-bath.

For heating liquids in tanks, barrels, or other deep vessels, steam may be conducted through a straight pipe to the bottom of the liquid in which the steam condenses giving up its latent heat to the contents. This method is, of course, applicable only in cases where there is no objection to the admixture of the water from the condensed steam, and is, therefore, employed only for heating liquids but never for evaporation or distillation.

For all ordinary operations where steam-heat is utilized the

steam may be advantageously used under a *pressure* of four pounds or even less.

OTHER APPARATUS.

356. *Earthenware pots,* thoroughly glazed and acid proof, are indispensable. They can be procured of all sizes from 5 liters to 300 liters, or even larger. They are used for solution, filtration, precipitation, washing, crystallization, etc.

Wooden vessels (tubs and barrels) may be used for some special preparations made on a large scale.

Glass and porcelain precipitation jars and wide-mouthed bottles are employed for smaller operations, and for still smaller quantities Erlenmayer flasks and beakers.

357. Porcelain *casseroles,* with handles preferably of wood, are most serviceable when of about 1 liter's capacity. Porcelain or "white ware" *pitchers* may also be used to a considerable extent for transferring liquids from one vessel to another, but not so conveniently as casseroles for *hot* liquids.

Tinware *dippers* (well tinned) may be used for many purposes in the laboratory.

358. *Bottles* for both solids and liquids, glass-stoppered as well as others, are required in plenty. Glass-stoppered so-called "tincture bottles," for acids and other solutions, may be used as large as of 12 liters capacity with great advantage, if large quantities of the liquids are employed; but larger bottles are less convenient and more liable to breakage.

359. *Evaporation dishes* have been sufficiently described in Chapter VII, *crystallizers* in Chapter IX, and *mortars and pestles* in Chapter I.

Funnels for filtrations, for collecting and washing precipitates and crystals, and for other purposes have been described in the chapters discussing those operations, and *flasks, retorts,* beakers, *wash-bottles,* and other requisite glassware, including tubing and fittings, were also mentioned to a sufficient extent in preceding chapters. Some apparatus will be more specifically described in Part II as occasion requires.

Sieves, spatulas, scoops, spoons, strainer holders, iron stands, hammer and anvil, cork borer, files, tongs, knives, shears, and many other implements and tools are needed in laboratories.

The catalogues of dealers in chemical and pharmaceutical apparatus should be consulted for further information and details. Such catalogues are abundantly illustrated.

360. In the training laboratories of pharmaceutical and other technological schools it is customary to have large water-baths and sand-baths, drying closets, fume chambers, and balance room for general use by all students, and to issue to each student a set of apparatus for his individual use, and special apparatus as required.

361. *An outfit of apparatus* sufficient for the production of experimental quantities of the great majority of the preparations included in Part II of this volume, but not including preparations requiring special apparatus, may be as follows:

An iron stand with heavy base and three rings of different sizes (respectively of 75, 100 and 125 millimeters diameter) on the rod ("retort stand").

One copper water-bath, 150 millimeters diameter, with three rings, and with two opposite handles.

One Russia-iron dish, 150 millimeters diameter and 25 millimeters deep, for a sand-bath.

A gas burner (or, in its place, a coal oil burner or a spirit lamp).

One No. 4 or 5 mortar and pestle.

One or two mm porcelain evaporating dishes (Meisen or Berlin ware).

One or two graduated glass measures, on foot, each of about 200 Cc. capacity (metric glass "graduates").

Two or three thin glass beakers, of about 250 Cc. capacity.

One glass flask with wide neck of medium length and flat bottom, capacity 600 Cc.

One round bottom glass flask, 600 Cc.

One round glass (or porcelain) dish, about 150 millimeters diameter and 35 to 50 millimeters deep, for use as a crystallizer.

One 150 mm glass funnel, 60° angle, with long stem having beveled end.

One 100 mm glass funnel of same kind.

One Elenmeyer flask of 500 Cc. capacity.

One Elenmeyer flask of 300 Cc. capacity.

One Elenmeyer flask of 200 Cc. capacity.

One Elenmeyer flask of 100 Cc. capacity.
One Elenmeyer flask of 50 Cc. capacity.
One wide-mouthed 2-liter bottle.
Two wide-mouthed 1-liter bottles.
Two wide-mouthed half-liter bottles.
A dozen 150 mm test-tubes.
One double ended 150 mm horn spatula.
Half a dozen glass stirring rods, assorted sizes, 150 to 300 millimeters.
Two packages of coarse (rapid filtering) filter paper ("S. & S.," or Swedish, or French) to fit the 150 mm funnel.
One package of the same kind of filter paper to fit the 100 mm funnel.
Corks, tapering, XXX, extra long, assorted sizes.

This outfit will enable the student to make moderate quantities of nearly all the chemicals mentioned in this manual. For the remaining preparations a few additional pieces of apparatus are required, such as retorts, wash bottles, glass tubing, rubber tube connections, thistle tubes, perforated rubber stoppers, etc.

CHAPTER XX.

SOME LABORATORY RULES AND PRECAUTIONS, AND WHAT TO DO IN CASE OF CERTAIN ACCIDENTS

362. Order, system and **cleanliness** must prevail to the utmost possible extent.

But as the materials and products handled are generally such as would quickly ruin clothing, the laborant should wear *"overalls,"* or at least *apron* and *sleeves* of rubber cloth.

The laboratory floor, fixtures and furniture must be kept clean and free from dust.

Whenever any substance is spilled on a table or elsewhere the accident should receive immediate attention.

Especial care should be exercised in handling acids, alkalies, bromine, phosphorus, and other corrosive or inflammable substances.

Operations attended by the evolution of irritating or dangerous vapors should be performed out of doors, or under the hood or fume chamber. When such operations are performed outdoors the operator should place himself in such a position that the vapors or gases evolved are carried away from him by the air currents.

Reactions liable to be violent must be carefully performed, watched and controlled to prevent explosions, fire, breakage, spattering of corrosive or hot liquids, or the sudden evolution of large volumes of noxious gases.

Operations involving danger should never be undertaken except when necessary.

It must be remembered that powerful oxidizing agents and reducing agents generally react upon each other with great velocity frequently attended with explosion, fire, or other dangers.

363. Acids must be kept in a safe place and in strong containers not too large to be handled with convenience and with a minimum of risk.

Carboys of acids must not be moved about, but should have their permanent place on the ground or on a special stand in a

location where the least danger would result from their being broken. Earthenware vessels may be placed under the stand to catch escaping acids in case of breakage of a carboy. When bottles are filled from the carboys, special acid syphons of glass should be used in order that the carboy may be left undisturbed in its position and to avoid, as far as possible, all danger of spilling.

Whenever a carboy of acid is broken, immediate steps must be taken to prevent any greater destruction than is unavoidable. The escaped or escaping acid must be caught up in a jar or dish if possible, or neutralized as far as possible with sodium carbonate, which should be at all times kept near by for this purpose, and plenty of water should be turned or poured upon the acid to dilute it. In order that this may be done quickly and without hesitation or danger, the acid carboys should stand in a place where the floor is covered with asphalt and inclined toward an ample drain connected with the sewer and near the hydrant water.

364. Should the acid be *sulphuric acid,* remember that the addition of water will cause the generation of great heat and steam, and that the mixture will be liable to boil and spatter about, especially if the quantity of water added is comparatively limited. It is, therefore, best to allow the acid to run off unless it can be caught in a large earthenware pot or dish. Then soda must be used freely and a *copious* supply of water.

365. If *hydrochloric acid* escapes in large quantity from any broken vessel, throw plenty of sodium carbonate upon it, together with much water, and if the air gets filled with the irritating vapor scatter some ammonia water about the place so that the vapor of ammonia may neutralize that of the acid.

366. *Nitric acid* is more destructive than any other. It not only attacks with great vigor any organic matter with which it comes in contact, but, as it does so, it fills the air with suffocating red nitrous vapors. Fire and explosions are liable to be caused by the action of nitric acid on wood, especially if the wood is resinous. Sawdust which is so effective in absorbing ordinary liquids should never be thrown upon acids, especially nitric acid. When a large amount of nitric acid has escaped, ammonia should be freely used to neutralize the vapor while soda should also be utilized, and an abundance of water.

In accidents of this nature the fire hose should be used.

367. When small quantities of strong acids are spilled or scattered about, they should be immediately neutralized, diluted and wiped up. The eyes and clothing of laborants, and especially of careless and inexperienced beginners, are sometimes destroyed by acids. The greatest care should, therefore, be exercised whenever any strong acid is used, for even a drop of it may easily do very serious damage. A drop or two of concentrated acid on the floor, if stepped upon, will eat through the shoe leather; on the table it may not only make holes in the clothing, but may find its way to hands, face and eyes.

Dilute acids are also destructive; they must be cautiously handled and immediately neutralized and thoroughly washed off when spilled.

368. Strong acids should never be directly mixed with strong alkalies except in case of the escape of large quantities of the acid as described, and then only when plentiful additions of water are at once thrown upon the acid, and it is dangerous to be too near the spot where the acid and alkali are mixed or to face it, for the temperature of the reaction is sure to cause the liquid to spatter about.

When it is necessary in laboratory operations to mix a strong acid and an alkali both should be as diluted as the circumstances require, and they should be cautiously and *gradually* mixed in a capacious vessel.

369. Water must never be added to strong sulphuric acid for the result is liable to be disastrous from the violent reaction which may throw considerable portions of the acid out of the vessel in all directions. When it is necessary to dilute concentrated sulphuric acid the acid must be slowly added to the water as described in the proper place in this book. The addition of water-solutions of alkalies, ammonia, alkali carbonates, and some other compounds of the alkali metals is even more dangerous than the addition of water alone to strong sulphuric acid. All water-solutions and also alcoholic liquids act in a similar manner when added to concentrated sulphuric acid.

370. Stock bottles containing acids must be strong glass-stoppered bottles, and not too large.

Bottles containing volatile acids, such as nitric acid, hydrochloric acid and acetic acid, must not be entirely filled unless kept in a very cool place, and when the bottle is more than half filled

with such an acid the contents should be cool when the bottle is opened and the operator's face should be turned away, because if the acid is not cool there may be, when the stopper is removed, an out-rush of acid vapor great enough to do injury to the eyes and the respiratory organs.

371. All acids should be kept in a cool place.

372. When a drop or more of dilute acid accidentally gets on the clothing a little dilute ammonia should be applied.

373. Alkalies are not so dangerous as the strong mineral acids, but they are corrosive enough to do a great deal of damage if allowed to come in contact with organic matter. They destroy clothing, and eat into wood. When strong lye is spilled it should be at once diluted with plenty of water, the dilute liquid wiped up, very dilute sulphuric or hydrochloric acid next applied, and finally more water.

374. Bottles containing solutions of potassium hydroxide, sodium hydroxide or ammonia must be strong glass-stoppered bottles; and bottles containing ammonia solution must not be entirely filled, must be cool when opened, and opened cautiously, the face of the operator being averted as in the case of opening bottles containing volatile acids. Loss of eyesight has resulted from opening filled and not sufficiently cool bottles of strong ammonia water owing to the great rush of gaseous ammonia from the container.

375. When solid KOH or NaOH is to be dissolved in water it is best to add the alkali to all of the water, and to add it gradually if the solution is to be a concentrated one. Great evolution of heat attends the solution of the strong alkalies in water, probably due to condensation caused by molecular combination.

376. The glass stoppers of bottles containing solutions of KOH or NaOH must be coated with "petrolatum." Unless this is attended to it usually happens that the stopper becomes so firmly cemented to the neck of the bottle by the chemical action of the alkali upon the ground surfaces of the glass that the removal of the stopper is impossible and the neck of the bottle must be cut or broken off in order to recover the contents.

377. Bromine is a terrible substance to deal with if allowed to escape. It rapidly evaporates and fills the room with its vapor, which is so irritating and destructive that it can not be inhaled without grave danger, and it attacks the eyes severely. It also

attacks clothing, and the liquid bromine even eats into wood very quickly.

Bromine is necessarily kept in comparatively small, strong, glass-stoppered bottles, but the stoppers usually become fast in the necks of the bottles so that their removal is in some cases impracticable. It is then necessary to cut or break the neck off, or to break the bottle itself, to empty it when the bromine is to be used. This is, of course, a hazardous operation unless the operator is expert or careful and takes such precautions as will prevent the liability to serious results. When the bromine bottle is to be opened it should be held over and near the bottom of a strong porcelain dish, and this in turn should be placed in a second strong porcelain dish. At the same time an empty glass stoppered bottle with a small funnel already placed in its neck should be at hand into which the bromine can be at once transferred from the original bottle or from the porcelain dish should the original bottle be broken to such an extent that the bromine runs out into the dish. The whole operation should be performed either outdoors, or under the fume hood, or on the ledge outside the window.

Whenever the bromine is to be used *with water* for some laboratory operation, as, for instance, in making bromide if iron, it is best to open the bottle of bromine over a porcelain dish containing water, or to break the bottle in the body of the water in the dish. The weight of the water must then be known before the bromine is added, or the gross weight of bromine and bottle, or both, and the tare of the bromine bottle found in some way. The bromine and water can be transferred from the dish to a tared flask or some other vessel and there weighed, the fragments of the bottle remaining in the dish.

The neck of a bottle is best broken off by first filing a deep groove around it and then striking it gently.

When large quantities of bromine are used the bottles should be put in a safe, cool place, preferably out in the yard and buried in a mound of sand or clay by which the bromine might be partially absorbed should any bottle be broken.

In case of the escape of bromine in the room a liberal quantity of sodium carbonate or potassium carbonate should be as quickly as possible thrown upon it after which plenty of water should be added. Ammonia water should be scattered on the

floor near the bromine in order that the vapors of the H_3N and the Br_2 may neutralize each other.

If liquid bromine is allowed to act upon any part of the body it may quickly produce serious wounds which are not only very painful but quite difficult to heal.

The vapor attacks the eyes and face and the respiratory organs. Should the effects be so severe as to require alleviation a little ammonia vapor may be *very cautiously* inhaled from diluted ammonia water, and the face and eyes freely bathed with cold water.

378. Phosphorus, and also the metals potassium and sodium, are fire-dangerous.

Phosphorus must be kept in water contained in glass-stoppered bottles in a safe place. When required for use it must be handled and divided in water or in a moist condition, and it can not be with safety wiped dry for weighing. Should it ignite it must be at once thrown into water. If ignited while held in the hand or between the fingers it may cause severe wounds.

Potassium and *sodium* must be kept in a liquid hydrocarbon in tightly stoppered bottles. They ignite when exposed to the air or come in contact with water.

379. *Ether,* petroleum spirit (benzin) and other **inflammable liquids** must be kept in strong, tightly closed containers (not filled) in a cool place, away from any light or fire, and when required for use they should not be brought near any flame. It must always be remembered that the vapors of inflammable volatile liquids may be conveyed through and with the air and become ignited by a flame a considerable distance from the vessel containing the liquids. Extraordinary precautions must be taken to prevent fire when any distillation or other operation is performed in which a volatile inflammable liquid is heated. All such operations should be performed on the fire table near the sink and in such manner that the inflammable vapor can not come in contact with the flame indirectly or directly supplying the heat.

Inflammable vapors form explosive mixtures with air.

380. Fire in a laboratory may be caused not only by strong acids, phosphorus, ether and other inflammables and explosives, but more easily than elsewhere from the careless handling of matches and heating apparatus, because heat is so constantly utilized. Strict discipline in this direction is accordingly imperative.

Matches must be kept in a safe place. Gas should never be left burning except when necessary. Water-baths should never be permitted to become empty while in use.

Whenever the laboratory is about to be closed for the night, or temporarily deserted at any time, a careful inspection must be made to see that no flame of any kind is left burning and no operation continued which is liable to prove dangerous or to cause any injury or loss by fire, explosion, or any damage to materials, or to products in process of preparation.

In the training laboratories of technical schools the employment of common large sand-baths and water-baths for the use of the classes diminishes the liability to danger and damage by rendering unnecessary the constant use of gas burners as well as by substituting the far safer baths for many operations in which inflammable substances are employed.

Operations and experiments involving danger can be entirely avoided in college laboratories, and the risks of accidents be thus reduced to such as may happen anywhere as the result of gross carelessness or stupidity.

381. In all laboratory work the operators should keep a **journal** or record of every operation and of each important step in it. To save time it is generally advantageous or even necessary to keep two, three or even more preparations under way concurrently.

All vessels containing either materials, unfinished products, or finished products must be **labeled** so explicitly that no mistake can be made. To leave any substance, or mixture, or solution, unlabeled, trusting to memory to know what it is, must be regarded as inadmissible in a laboratory. Nothing should be left unlabeled for even an hour. The label on the vessel should not only indicate plainly what it contains but what the contents are to be used for, the date, and also the name of the laborant having the matter in hand.

Notes should be made in the laboratory journal of all preparations made, date when commenced, materials used and quantities of them, method adopted (if there is a choice), any interesting, instructive or important observations made in regard to the results, the date on which the preparation is finished, the quantity of product obtained and its character or quality, and what was done with the bye-product, if anything.

382. Unnecessary delay in finishing the operations undertaken in the laboratory is very liable to cause loss or waste of materials as well as of the time and labor already bestown. This applies to analytical processes as well as to the production of chemical preparations. No preparation should be started at an hour when it would be impracticable to give it the attention necessary to carry the process up to a point at which further attention may be deferred without disadvantage or risk.

Unfinished products, especially if moist, are generally more liable to decomposition or damage from exposure than finished preparations.

383. How to clean apparatus. All laboratory apparatus and implements, as well as the furniture, must be kept clean. Operators who do not clean their apparatus and utensils thoroughly and keep them in perfect order and ready for use, and who leave the table in disorder, soiled or wet, are wholly unfit laborants. Every piece of apparatus should, after using it, be as thoroughly cleaned as any dish, plate or tumbler set before properly fastidious persons at the dinner table.

But chemical apparatus, and especially mortars and pestles, require at times chemical agencies to clean them, particularly if they have been permitted to remain long in contact with substances which act upon them chemically or which are absorbed.

"Dirt is only misplaced matter," and even pharmacists and chemists are apt to recognize or notice only colored dirt. But white or colorless dirt is quite as objectionable and sometimes more so than the more obtrusive kinds. Hence all chemical and pharmaceutical apparatus must be scrupulously cleaned and rinsed.

Insoluble substances rubbed into the pores of mortars and pestles or adhering to glass and glazed surfaces should be converted into soluble substances to facilitate their removal. Thus iron stains may be removed with the aid of nitric acid or hydrochloric acid, or, if necessary, both together; insoluble mercury compounds are removed with nitric acid; antimony compounds with *strong* hydrochloric acid; iodine stains with potash solution; organic extractive with strong potash solution; arsenic and its compounds with strong nitric acid; silver compounds with strong ammonia or with nitric acid, or the two used alternately; lead compounds with nitric acid, etc.

The piece of apparatus cleaned may be known to be free from the substances sought to be removed when their presence can no longer be detected by means of sensitive reagents. To simply turn colored dirt into colorless dirt by means of chemicals is not to remove it; invisible dirt, if not removed, can usually be easily enough rendered visible by reagents.

PART II.

LABORATORY MANUAL OF INORGANIC CHEMICAL PREPARATIONS.

LABORATORY MANUAL.

INTRODUCTORY.

The chemical products for which methods of preparation are here given include all of the inorganic compounds commonly employed in every day life, in the chemical arts and industries, and in medicine and pharmacy, together with many interesting and instructive preparations which are not much used. The inorganic preparations of the pharmacopœias are all included.

Processes attended with great danger or difficulty, and such as require expensive and unusual apparatus, have been omitted.

The nomenclature is, as will be seen, a technical English nomenclature, and the corresponding latinic titles have been given a subordinate position, together with the most common non-technical names and officinal designations.

The proportions and quantities specified in the formulas are as a rule reduced to the simplest terms and the method of expressing the proportions in "parts by weight" has been very largely employed; but when circumstances seemed to require it the quantities of solids have been expressed in Grams (Gm) and those of liquids in milliliters (ml).

In large practice laboratories it is rarely practicable to weigh all liquids, while it is very easy to measure them in graduated glass measures; and in most cases the quantities required can be measured with sufficient accuracy to insure entirely satisfactory results. Students can readily find the number of milliliters corresponding to any given number of Gm by dividing the number of Gm by the specific weight or multiplying that number by the specific volume (which is the reciprocal of the specific weight). Quantities of liquids below 5 ml or above 500 ml should be weighed instead of being measured.

When these formulas are employed in college laboratories for practice in preparation work it is suggested that the lessons be

to some extent so selected that the product obtained from each may serve as one of the materials for the next lesson. The student may, for instance, first be given the iron and sulphuric acid to make ferrous sulphate; he may then make not only ferrous sulphate in large crystals, but also turbidated and precipitated ferrous sulphate, dried ferrous sulphate, ferric sulphate, ferric subsulphate, ferric hydroxide, ferric pyrophosphate, ferric citrate, etc. He may be furnished the materials for the preparation of sodium phosphate and then make several phosphates and pyrophosphates from the same materials. It is also useful to require students to recover the bye-products wherever practicable, and to account for the materials furnished them by producing as large a yield of each product as may be practically obtainable.

The directions and notes are sufficiently explicit to enable individual students as well as other laborants to do the work successfully and saticfactory results are certainly unattainable without such explanations as can not be repeated to each individual student.

The working-formulas included in this work are applicable not only in the practice laboratories of technical schools, but also in the laboratories of manufacturing pharmacists and chemists.

WEIGHTS AND MEASURES.

All weights and measures in use in the United States of America are derived from the metric prototypes in the custody of the Office of Standard Weights and Measures at Washington. The prototypes referred to are the U. S. National Prototype Standard Meter and the U. S. National Prototype Standard Kilogram, both made of iridium-platinum and furnished to the American Government by the International Bureau of Weights and Measures in 1890.

The old customary weights and measures, as well as those of the metric system, are based upon and adjusted to the material metric standards mentioned.

THE METRIC SYSTEM.

The **meter** is the length of iridium-platinum prototype meter at Washington.

1 meter is equal to 10 decimeters.

1 meter is equal to 100 centimeters.

1 meter is equal to 1000 millimeters (mm).

The **kilogram** is the mass of the iridium-platinum prototype kilogram at Washington.

1 kilogram is equal to 1000 grams (Gm).

1 gram is equal to 1000 milligrams (mGm).

The **liter** is the volume of one kilogram of pure water at 4° C. *in vacuo*.

1 liter is equal to 1000 milliliters (ml).

[*Theoretically* the volume of 1 kilogram of pure water at 4° C. *in vacuo* is 1 cubic-decimeter; but the liter in actual use, being always obtained by weight, can not consistently be called a cubic-decimeter, especially as it seems probable that the actual liter measures less than a cubic-decimeter.]

1 cubic-decimeter is equal to 1000 cubic-centimeters (Cc.).

1 American yard is $\frac{3600}{3937}$ meter.

1 American commercial pound is $\frac{700000000}{154323563}$ kilogram.

1 American liquid gallon is the volume of 3785.434 Gm of pure water at 4° C. *in vacuo*.

WATER.

AQUA.

$H_2O=18$.

The Pharmacopœia of the United States defines "aqua" as "natural water in its purest attainable state." It must be colorless, clear, inodorous, and tasteless. Should not contain more than 100 milligrams of fixed impurities in one liter, and not more than traces of organic matter.

Spring water usually contains inorganic salts, consisting chiefly of the chlorides, sulphates and carbonates of calcium, magnesium, etc. Such water is called *hard water,* because when used with soap it produces insoluble compounds, which cause a sense of harshness. Hard water can be rendered less so by boiling it, because the calcium and magnesium carbonates, which are held in solution by the carbonic acid present, deposit as soon as that acid is expelled by the heat. Spring water is generally unfit for pharmaceutical uses on account of the mineral impurities it contains.

Well water often contains organic matter derived from sewage, etc., especially in thickly inhabited places, and must not be used unless careful examination shows it to be sufficiently pure. Water contaminated with organic substances contains ammonia. The presence of organic matter in sufficient quantity to impart odor to the water should at once condemn it. Where pure water is not obtainable for ordinary purposes, small quantities of organic impurities may be partially removed by adding a little alum; the ammonia and carbonates present cause the precipitation of aluminum hydrate, which, as it settles, carries other impurities with it.

Rain water is pure if collected in clean vessels as it descends from the clouds after the rain has continued long enough to purify the atmosphere from dust, etc. Falling off roofs and collected from the conduits in the usual way it is rarely pure.

Ice, when melted, affords a comparatively pure water.

River and lake water, containing usually but small amounts of calcium and magnesium salts, is called *soft water,* and is frequently pure enough for most purposes; it may, however, contain both organic and inorganic impurities.

Uses. Water is indispensable in medicine, pharmacy, chemistry, as well as in the household economy. It is the best solvent and diluent we have, being abundant, and capable of dissolving a great variety of substances. It is, moreover, chemically indifferent or neutral to most substances, and hence affords a means of reducing water-soluble substances to a liquid condition without otherwise altering their properties, and can be freely used to facilitate many chemical reactions.

Tests. Mix 100 ml of the water with 10 ml of diluted sulphuric acid; heat to boiling; then add enough solution of potassium permanganate (1 part to 1000 parts of distilled water) to give to the mixture a distinct rose-red color; then boil for five minutes. The color will not be entirely destroyed by this treatment unless more than traces of organic matter be present.

DISTILLED WATER—AQUA DESTILLATA.

Water, a convenient quantity.

Put the water into a suitable still of copper or tinned iron, the still to be about two-thirds filled. Connect it with a suitable condenser, and distil. The first portion that passes over is to be rejected, being contaminated with impurities taken up in its passage through the apparatus, and containing also any carbonic acid or other volatile substances which may have been present in the water.

When only about one-fifth or one-sixth of the water remains in the still, the process is to be arrested in order to prevent possible contamination with products of decomposition of organic substances by overheating.

In large laboratories it is necessary that a special distilling apparatus be exclusively devoted to the preparation of distilled water.

The most effective and satisfactory plan is to use a small boiler of the simplest construction permanently placed in brick work over a fireplace with good draft. Before the water to be distilled is put in the boiler it may be advantageously treated, first

with a little potassium permanganate and then with alum. The first portion of the distillate should be rejected as long as it does not hold the necessary tests.

Distilled water may be kept in large bottles the necks of which are closed by *loose* plugs of pure cotton ("absorbent cotton") instead of glass stoppers.

Tests. Should yield no residue on evaporation. In applying the permanganate test described above to distilled water, the rose-red tint should not only remain after five minutes' boiling, but should not be entirely destroyed, even after ten hours' standing in a covered vessel subsequent to the boiling.

It should not be affected by test-solutions of barium chloride (*sulphates*), silver nitrate (*chlorides*), ammonium oxalate (*calcium*), or mercuric chloride with or without the subsequent addition of potassium carbonate (ammonia and ammonium compounds).

ACIDS.

Caution in the handling and use of acids has been urged in the chapter devoted to laboratory rules and precautions. The most suitable containers for constant use in the laboratory are the common glass stoppered "acid bottles" of about two liters capacity.

The removal of the glass stopper which has become fast in the neck of an acid bottle is attended with risk. Gently tapping the stopper with a wooden block or stick, warming the neck of the bottle with a cloth dipped in hot water or by cautiously rotating it while held over the flame of a gas burner or spirit lamp, and inverting the bottle in a vessel containing enough warm water to cover the whole bottle-neck, are among the best means of loosening the stopper sufficiently to render the application of the stopper wrench successful. A block of wood with a deep groove in the center is an effective stopper wrench. If the bottle is nearly filled and the acid volatile (as nitric, hydrochloric or acetic acid) and concentrated, the face should be turned away when the stopper is removed.

The acids of different manufacturers and different pharmacopœias are not of uniform strength. It is, therefore, important that the laborant should know the particular strength intended by the directions or formula followed, and the strength of the acid he actually employs in order that the quantities or proportions may be adjusted as the circumstances may require. The employment of acids which are stronger or weaker than those prescribed by the manual is often practicable if the quantities be modified accordingly; but in many cases the particular strength prescribed can not be altered without disadvantage or failure.

As the acids are so many and so extensively used, and as many metallic salts of the organic acids must necessarily be included in any such manual as this, all of the common acids, organic as well as inorganic, are placed together in alphabetical order under the general head of acids before the other compounds are described.

ACETIC ACID.

ACIDUM ACETICUM.

$HC_2H_3O_2$=60.

Formerly prepared by acetic fermentation of dilute alcohol, or of weak saccharine liquids. It is now made by dry distillation of wood. Oakwood billets are heated in closed sheet-iron cylinders, one of the numerous products being impure acetic acid, or *wood vinegar,* which is afterwards purified.

The impure acetic acid obtained by the destructive distillation of wood is called pyroligneous acid, and has a disagreeable smoky odor from the empyreumatic products contained in it. Even some of the better grades of acetic acid contain empyreumatic matters, and only chemically pure acid is entirely free from them. To get rid of these impurities the acid is treated with lime and soda, the acetates of calcium and sodium are freed from the tarry matters, and then decomposed by sulphuric or hydrochloric acid. A nearly pure acetate of sodium can thus be made from the impure acid, and by distilling a mixture of this sodium acetate with sulphuric acid a purer grade of acetic acid is obtained, which can be further purified by again neutralizing with sodium carbonate, decomposing the acetate with sulphuric acid, and distilling as before. A pure sodium acetate is finally obtained, from which pure acetic acid may be made as follows:

Sodium acetate	18 parts
Sulphuric acid	13 parts

Reduce the acetate to a coarse powder; introduce it into a distilling flask; warm the flask; connect it with a condenser and receiver; add the sulphuric acid through a funnel tube, a little at a time, and continue the distillation until 12 parts of distillate have been collected.

Test the product with volumetric test-solution of potassium hydroxide to determine its strength and then dilute as required.

Properties. A solution composed of 36 per cent of hydrogen acetate and 64 per cent of water.

A clear, colorless liquid, of a distinctly vinegar-like odor, a

purely acid taste, and a strongly acid reaction. Sp. gr. 1.048 at 15°C., compared with water at+4° C. as 1.000. Its specific volume is accordingly 0.954. Miscible in all proportions with water and alcohol, and wholly volatilized by heat.

The presence of empyreuma is at once detected by a smoky odor when the acid is neutralized with KOH, NaOH, or $KHCO_3$.

An acetic acid of from 77 to 80 per cent strength has the sp. w. of about 1.0748; weaker and stronger solutions of acetic acid have a lower specific weight. Hence the strength of acetic acid can not be determined otherwise than by actual assay.

Valuation. To neutralize 6 Gm of acetic acid containing 36 per cent of absolute $HC_2H_3O_2$ requires 36 ml of normal volumetric test-solution of potassium hydroxide . (Each ml required of the test-solution indicates 1 per cent of absolute acetic acid.) Phenolphtalein is the indicator usually employed.

Diluted Acetic Acid.

Acetic acid	100 Gm
Distilled water	500 Gm

Mix them.

Description.—Diluted acetic acid contains 6 per cent, by weight, of absolute acetic acid.

Specific gravity: about 1.008 at 15° C.

It corresponds, in properties, to Acetic Acid (see *Acidum Aceticum*), and should respond to the same tests of purity.

To neutralize 24 Gm of diluted acetic acid should require 24 ml of potassium hydroxide test-solution (each ml corresponding to 0.25 per cent of the absolute acid), phenolphtalein being used as indicator.

Glacial Acetic Acid.

Nearly or quite absolute acetic acid.

Description.—At or below 15° C. (59 F.) a crystalline solid; at higher temperatures a colorless liquid. When liquefied and as near as possible to 15° C. it has the sp. gr. 1.056—1,058. Its properties are similar to those of acetic acid, and it is similarly affected by reagents.

To neutralize 3 Gm of glacial acetic acid should require not less than 49.5 ml of normal solution of potassium hydroxide (corresponding to at least 99 per cent of absolute acetic acid).

At low temperatures it crystallizes into an ice-like mass, and when it only partially congeals it has the appearance of a supersaturated solution.

When glacial acetic acid is diluted with water its specific gravity increases with a simultaneous fall in temperature until the mixture contains about 77 to 80 per cent of hydrogen acetate. Upon the further addition of water, however, the specific gravity decreases and the temperature of the mixture rises. An acetic acid of 47 per cent strength has the same specific gravity as the official glacial acetic acid, which is more than twice as strong.

The corrosive nature of glacial acetic acid renders it necessary to be very cautious in handling it, especially in removing the stopper from a full bottle which has been standing in a warm place, as vapors of the acid are liable to issue from the bottle with a rush at the moment the stopper is removed.

Glacial acetic acid is a remarkably effective solvent for resins, volatile oils, glucosides, alkaloids, and many other organic substances. It also dissolves normal bismuth nitrate.

BENZOIC ACID.

ACIDUM BENZOICUM.

$HC_7H_5O_2 = 122$.

Benzoic acid is contained in benzoin to the extent of from 10 to 19 per cent and may be obtained by sublimation. The benzoin is coarsely powdered and spread in a thin layer on a flat tinned iron pan. This pan, for 500 Gm of benzoin, ought to be about 300 mm in diameter and about 35 mm deep. A sheet of porous paper is fastened over the pan, after which a cone or dome of thick paper is tied securely around the edges of the pan. The apparatus is then placed on an iron plate, covered with a thin layer of sand. Heat is now applied, gradually, until the odor of benzoic acid becomes quite noticeable through the paper, but the temperature must not be raised too high, for the product will then become fused and discolored.

Benzoic acid can also be made by the wet way: boiling benzoin with calcium or sodium hydroxide, and decomposing the benzoate of calcium or sodium with hydrochloric acid. Sodium carbonate may also be used:

Benzoin	4 parts
Sodium carbonate	1 part
Hydrochloric acid, sufficient.	

Digest the benzoin with the sodium carbonate, previously dissolved in 10 parts of water, for three hours at about 60° C. Then boil the mixture a few minutes, filter, and neutralize with hydrochloric acid. Collect the precipitated benzoic acid, and purify it by dissolving it in 20 times its weight of boiling water. digesting the solution with a little animal charcoal, filtering, concentrating the solution by evaporation, and crystallizing.

Benzoic acid is now manufactured chiefly from toluol; but also from hippuric acid and from naphthalin. This artificial benzoic acid is generally pure, chemically, but differs from pure natural benzoic acid by being more compact and inodorous. Natural benzoic acid obtained from benzoin by sublimation, is very soft and bulky, and is fragrant. Benzoic acid prepared from benzoin by the wet process is, however, scarcely fragrant. The agreeable odor of sublimed benzoic acid seems to be due to ethyl benzoate, or to some volatile oil, or both, existing in the resin and accompanying the acid when sublimed. The fragrant benzoic acid prepared from benzoin by sublimation is the only kind prescribed by several pharmacopœias.

In the trade the natural benzoic acid is styled as "English," and the artificial as "German Benzoic Acid."

Benzoic acid has marked antiseptic properties, and a solution of one part of benzoic acid and one part of borax in 100 parts of water is often employed. The borax aids the solution of the benzoic acid.

Benzoic acid must be kept in tightly closed bottles, in a cool place, and well protected against light.

Description.—*Natural benzoic acid* (that prepared from Siam benzoin by sublimation) is white or yellowish white, in scales or needles of silky lustre, and has an agreeable benzoin-like odor and a somewhat pungent taste. Melts at 120°.

Artificial benzoic acid is white, consists of lustrous needles, is odorless, and has a warm, acid taste. Melts at 121.°4.

Benzoic acid is soluble at 15° in about 400 parts of water, and in 2 parts of alcohol; in 17 parts of boiling water, and in 1 part of boiling alcohol; also in 2.5 to 3 parts of ether and in 10 parts of glycerin. It is also soluble in fixed and in volatile oils.

BORIC ACID.

ACIDUM BORICUM.

$H_3BO_3=62.$

Borax, in powder	100 parts
Nitric acid (63%)	58 parts
Distilled water.	

Dissolve the borax in 250 parts of boiling distilled water, and filter while hot. Add the acid to the hot filtrate and stir. Set aside in a cold place for a day.

Collect the crystals on a muslin or paper filter and wash them with a small quantity of cold distilled water.

Re-dissolve the washed crystals in 200 parts of boiling distilled water, and set the solution aside until quite cold.

Place a loose layer of clean cotton in the throat of a glass funnel and collect the crystals upon it. Drain thoroughly. Transfer the product to a dry muslin cloth or a sheet of bibulous white paper, spread it out, and dry it in a moderately warm place.

Reaction. $Na_2B_4O_7.10H_2O+2HNO_3=4H_3BO_3+2NaNO_3+5H_2O.$

Notes. It will be seen that 381 parts of borax requires 200 parts of nitric acid of 63 per cent strength. A slight excess of acid is, however, necessary; hence 58 parts is ordered instead of 55 for 100 parts of borax. Nitric acid is preferred to hydrochloric acid because sodium nitrate is more readily soluble than sodium chloride. Sulphuric acid is not employed because it is not so readily washed out.

The yield is theoretically about two-thirds of the weight of

the borax used, but only about 50 to 60 per cent can be obtained in practice.

Description.—Colorless or white, pearly, lamellar crystals, somewhat unctuous to the touch; taste feebly acid, slightly bitterish. Boric acid dissolves in 30 parts of water at 15° C.; in 26 parts at 19°; 15 parts at 25°; about 4.75 parts at 75°; 3.55 parts at 87.°5; and in 2.97 parts of boiling water.

Or, 100 parts of water dissolves 3.9 parts of boric acid at 19°; 6.8 parts at 25°; 9.8 parts at 50°; 21 parts at 75°; 28 parts at 87.°5; and 34 parts at 100°.

A saturated water-solution of boric acid at 19° contains 3.75%; at 25° it contains 6.27%; and at the boiling point of water 25.17 per cent.

It is soluble in 15 parts of alcohol at 15° according to the U.S.P. (According to Wittstein it is soluble in 6 parts of alcohol; but according to the British Pharmacopœia it requires 30 parts of alcohol of 90 per cent strength.)

It is soluble in about 4 to 5 parts of glycerin at 15° (in 10 parts according to the U.S.P.).

Crystallized Boric Acid.

Boric acid may be obtained in rather large, handsome, pearly crystals by the spontaneous evaporation of a filtered solution made of 1 part of boric acid and 20 parts of hot distilled water. This solution should be slowly cooled.

Handsome crystals may also be obtained by the spontaneous evaporation of a saturated alcoholic solution.

Boroglycerin.

(GLYCERYL BORATE. BOROGLYCERIDE.)

Boric acid, in fine powder.....	2 parts
Glycerin	3 parts

Heat together in a porcelain dish at about 150° C., stirring well, until aqueous vapors cease to be given off and a homogeneous, transparent mass is formed, which becomes firm and tough on cooling.

Reaction. $C_3H_5(OH)_3+H_3BO_3=C_3H_5BO_3+3H_2O$.

Notes. If the materials are pure, and if no foreign organic matter be permitted to get into the dish, the product will be perfectly clear and colorless. The heat applied should not be greater than necessary. Boroglycerin is a remarkably tenacious substance, which, while warm, can be drawn into slender threads of great length. It is readily soluble in water, in alcohol, and in glycerin.

Glyceritum Boroglycerini; U.S.

A glycerin-solution of glyceryl borate (boroglycerin or boroglyceride) is ordered by the Pharmacopœia, prepared as follows:

Boric acid, in fine powder..... 31 Gm
Glycerin.

Heat 46 Gm of glycerin in a tared porcelain dish to not over 150° C.; add the boric acid gradually and stir well. When all of the boric acid has been added and dissolved, continue the heat, stirring constantly to prevent the formation of any film on the surface of the mixture, until the weight of the contents of the dish shall have been reduced to 50 Gm. Then add 50 Gm of glycerin, mix well, and transfer the product to a suitable container, which must be tightly closed.

Notes. The preparation is a perfectly clear, colorless, thick liquid. It is hygroscopic.

CITRIC ACID.

ACIDUM CITRICUM.

$H_3C_6H_5O_7.H_2O=210$.

Contained in various plant juices, especially of lemons, limes, and currants. The juice is allowed to ferment enough to decompose the sugar. Then follows clarification with albumen, after which the juice is treated with prepared chalk, and the calcium citrate decomposed with sulphuric acid:

Lemon juice..... 800 ml
Prepared chalk..... 45 Gm
Sulphuric acid..... 25 ml

Heat the lemon juice to the boiling point, and gradually add the chalk until it no longer causes effervescence. Collect the deposit on a muslin strainer and wash it with hot water till the filtered liquid passes colorless. Mix the deposited calcium citrate with 200 ml of water, and gradually add the sulphuric acid previously diluted with 300 ml of water. Boil gently for half an hour, keeping the mixture constantly stirred. Separate the acid solution by filtration, wash the insoluble matter with a little distilled water, and add the washings to the solution. Concentrate this solution to the density of 1.21, then allow it to cool, and after twenty-four hours decant the liquor from the crystals of calcium sulphate which have formed, continue the evaporation of the solution until a pellicle forms, and then set aside to cool and crystallize. If necessary, purify the acid by recrystallization.

Reaction. The free citric acid in the lemon juice is neutralized by the calcium carbonate, whereby calcium citrate is formed:

$$2(H_3C_6H_5O_7.H_2O)+3CaCO_3=Ca_3(C_6H_5O_7)_2+5H_2O+3CO_2.$$

When the calcium citrate is treated with sulphuric acid, the following reaction occurs:

$$Ca_3(C_6H_5O_7)_2+3H_2SO_4=2H_3C_6H_5O_7+3CaSO_4.$$

Notes. A small amount of acid calcium citrate always remains in the liquid after the treatment with chalk. This acid salt prevents the precipitation of coloring matter, and after the removal of the neutral calcium citrate, the acid citrate can be decomposed by milk of lime, yielding normal citrate.

The precipitated calcium citrate is washed with warm water until the washings are nearly colorless. After cooling, it is decomposed by sulphuric acid added in excess to prevent the formation of acid citrate, which would prevent crystallization of the citric acid. The calcium sulphate is washed and then thrown away; but the last of the calcium sulphate does not separate until the solution has been evaporated down to 1.21 sp. gr. The final evaporation to crystallization must not be carried too far, as the free sulphuric acid present would then blacken the product when sufficiently concentrated to decompose the citric acid.

Recrystallization is necessary to render the crystals colorless

and pure. Sometimes the solution must be decolorized by animal charcoal.

Description.—Colorless, translucent, prisms; odorless; having an agreeable, purely acid taste; efflorescent in warm air, and deliquescent when exposed to moist air.

Soluble, at 15° C., in 0.63 part of water, and in 1.61 parts of alcohol; in about 0.4 part of boiling water, and in 1.43 parts of boiling alcohol; also soluble in 18 parts of ether.

HYDRIODIC ACID.

ACIDUM HYDRIODICUM.

HI=127.5.

A solution of hydriodic acid, or "diluted hydriodic acid," containing 10 per cent of hydrogen iodide, may be prepared by a process similar to that prescribed by the Pharmacopœia for making the syrup with the exception that water is added instead of the same quantity of syrup, at the end.

Diluted hydriodic acid is a colorless, odorless liquid of acid taste. It should be kept in glass-stoppered bottles, entirely filled and placed in a cool, dark place. It is very unstable; but may be preserved for a time with the aid of sugar.

The official

Syrup of Hydriodic Acid

is made as follows:

Potassium iodide	13 Gm
Potassium hypophosphite	1 Gm
Tartaric acid	12 Gm
Water	15 ml
Diluted alcohol.	
Syrup.	

Dissolve the two potassium salts in water, and the tartaric acid in 25 ml of diluted alcohol. Mix the two solutions in a vial, shake it thoroughly, and place it in ice-water for half an hour, occasionally shaking. Then filter the mixture through a small,

rapidly-acting, white filter, and carefully wash the vial and filter with diluted alcohol, until the filtrate ceases to produce more than a faint cloudiness when a drop or two is allowed to fall into silver nitrate test-solution. Reduce the filtrate, by evaporation in a tared capsule, on a water-bath, to 50 Gm, and mix it, when cold, with enough syrup to make the product weight 1000 Gm.

Keep it in small filled glass-stoppered bottles in a cool, dark place.

Notes. The hypophosphite is added to form some hypophosphorous acid which tends to preserve the product.

Description.—A clear, colorless, odorless, syrupy liquid containing about 1 per cent by weight of absolute hydrogen iodide, or about 1.3 Gm in each 100 ml. Its sp. w. is about 1.313.

When it becomes discolored from free iodine it should be rejected. A darker color than a pale straw is inadmissible.

DILUTED HYDROBROMIC ACID.

ACIDUM HYDROBROMICUM DILUTUM.

HBr=81.

Sulphuric acid	14 parts
Potassium bromide	12 parts
Distilled water, sufficient.	

Put two parts of the water in a porcelain dish capable of holding about 32 parts; stir the water into a rapid rotary motion; then add the sulphuric acid slowly and in a thin stream, continuing the stirring with a glass rod.

Put the potassium bromide, previously reduced to coarse powder, in the dish; add 13 parts of water, heat the mixture by a sand-bath until the salt is dissolved, and while the solution is still hot add to it the sulphuric acid mixture, stirring briskly. Set aside for twenty-four hours.

Decant the acid supernatant liquid (hydrobromic acid) from the crystals of potassium sulphate, and pour it into a suitable distilling flask. Wash the crystals with a little cold water, and add these washings to the liquid.

Provide the flask with a perforated stopper and safety tube;

connect it with a well cooled receiver, and distill nearly to dryness. Assay the distillate and dilute it with enough distilled water to make the final product contain 10 per cent of hydrogen bromide.

Reaction. $2KBr+H_2SO_4=K_2SO_4+2HBr$.

Other Methods.

Hydrobromic acid may also be made from bromine and hydrogen sulphide as described under the title of Ammonium Bromide, and from potassium bromide with tartaric acid as follows:

Potassium bromide	4 parts
Tartaric acid	5 parts
Distilled water	25 parts

Dissolve the bromide and the acid, separately, each in 12.50 parts of distilled water. Filter the solutions. Mix them, stirring well. Filter out the cream of tartar. Assay the solution and adjust its strength to 10 per cent.

Valuation. To neutralize 8.08 Gm of diluted hydrobromic acid (10 per cent of HBr) requires 10 ml of normal solution of potassium hydroxide. Each ml of the volumetric test solution corresponds to 1 per cent of hydrogen bromide. Phenolphtalein is the indicator used.

One ml of normal solution of KOH is the equivalent of 0.08076 Gm of absolute HBr.

Description.—A clear, colorless, odorless, strongly acid liquid having a sp. w. of about 1.077 at 15°.

It is to be kept in glass-stoppered bottles in a cool, dark place.

HYDROCHLORIC ACID.

ACIDUM HYDROCHLORICUM.

$HCl=36.4$.

Hydrochloric acid is prepared by double decomposition between sodium chloride and sulphuric acid:

$NaCl+H_2SO_4=NaHSO_4+HCl$; and

$NaCl+NaHSO_4=Na_2SO_4+HCl$.

The hydrochloric acid of the Pharmacopœia of the United States contains 31.9 per cent of HCl and has a sp. w. of about 1.163 at 15°. It is a colorless, corrosive, fuming liquid, of suffocating acid odor, and intensely acid taste and reaction. When diluted with twice its volume of water the acid ceases to give off fumes and becomes odorless.

The acid styled "C.P." in the trade usually holds the pharmacopœical tests as to purity.

The customary "five-pint-acid-bottles" hold six pounds of hydrochloric acid.

The white vapors given off by strong hydrochloric acid are due to the difference in the proportion of moisture contained in the air and in the acid as well as to the formation of ammonium chloride when the acid vapors meet ammonia in the atmosphere.

Valuation. To neutralize 3.64 Gm of official hydrochloric acid, diluted with 10 ml of water, should require 31.9 ml of normal solution of potassium hydroxide. Each ml of this volumetric solution corresponds to 1 per cent of absolute HCl. Phenolphtalein is used as the indicator.

One ml of normal solution of potassium hydroxide is the equivalent of 0.03637 Gm of absolute HCl.

Action on metals. Dissolves iron and zinc readily; it also acts on nickel and aluminum, and warm acid attacks tin. Hydrochloric acid does not attack platinum, gold, silver, mercury, lead, copper, arsenic, antimony and bismuth.

Diluted Hydrochloric Acid.

Hydrochloric acid	100 Gm
Distilled water	219 Gm

Mix them. Keep the product in glass-stoppered bottles.

Diluted hydrochloric acid contains 10 per cent of hydrogen chloride.

Specific gravity: about 1.050 at 15° C.

It does not fume in the air, and is without odor, but otherwise it corresponds in properties to hydrochloric acid.

To neutralize 3.64 Gm of diluted hydrochloric acid should re-

quire 10 ml of normal potassium hydroxide solution (each ml corresponding to 1 per cent of the absolute acid), phenolphtalein being used as indicator.

DILUTED HYDROCYANIC ACID.

ACIDUM HYDROCYANICUM DILUTUM.

HCN=27.

Must contain just 2 per cent of hydrogen cyanide:

Potassium ferrocyanide, in coarse powder.... 4 parts
Sulphuric acid ..... 3 parts

The apparatus necessary consists of a long-necked flask fitted with a twice perforated stopper carrying a safety tube and the bent tube connecting the flask with a Liebig's or other suitable condenser, the exit tube of which should dip into the liquor in the receiver. The receiver should be tared, should be large enough to permit the final adjustment of strength without a change of vessel, and should be placed in broken ice or ice-water.

Put 16 parts of distilled water in the receiver.

The ferrocyanide is to be dissolved in 20 parts of water and then introduced into the flask. The acid is diluted by adding it gradually to twice its weight of water, and, after connecting the whole apparatus together properly and making all the joints tight, the cold diluted acid is poured in through the safety tube. Heat the flask on a sand-bath to the boiling point, and continue applying a moderate heat until there is but little liquid mixed with the saline mass remaining in the flask. Then detach the receiver and assay a sufficient portion of the contents. The distillate is then to be diluted with distilled water so that the finished product will have the required strength (2 per cent).

Reaction. $2K_4FeCy_6+3H_2SO_4=3K_2SO_4+K_2Fe_2Cy_6+6HCy.$

Notes. A flask is used instead of a retort because more convenient. If a retort be used it should be directed upward to prevent contamination of the distillate from spurting.

The heat required is not far above the boiling point of water.

A sand-bath is to be preferred to naked flame to avoid bumping. The reaction should be slow and regular, which requires rather diluted solutions, as here indicated, and sufficient control of the heat. But too great dilution favors bumping.

When the process is carried out as directed in the formula above stated, the distillate will contain about 3 to 4 per cent of hydrocyanic acid.

The official method of ascertaining the strength of the distillate is as follows:

Mix 0.27 Gm of the distillate in a flask of 100 Cc. capacity with sufficient water and magnesia to make an opaque mixture of about 10 Cc. Now add two or three drops of potassium chromate test solution, and then run in from a burette a quantity of decinormal volumetric silver nitrate solution just sufficient to produce a red tint, which does not disappear on shaking. Each ml used of the silver solution corresponds to 1 per cent of absolute hydrocyanic acid.

Multiply the per cent thus found by the total weight of the distillate in grams and divide the product by 2; the quotient shows the total grams of 2 per cent acid which can be made from the whole amount of the distillate. Therefore, the difference between the weight of the distillate obtained and the weight of the finished product which it will make will be the quantity of distilled water to be added.

For example, if we have 837 Gm of distillate of 3.6 per cent strength we would add 669.6 Gm of distilled water to the 837 Gm of distillate, thus obtaining 1506.6 Gm of final product, because—

$$\frac{3.6 \times 837}{2} = 1506.6.$$

After dilution the product should be again assayed, when 1.35 Gm of the preparation should require 10 ml of the decinormal volumetric silver nitrate solution for complete precipitation.

Diluted hydrocyanic acid always sustains loss of hydrogen cyanide in handling it. This weakening of the acid is appreciable in filling it in small bottles, and as it must be kept in small bottles to avoid too great and frequent exposure to air in using, it may be well to make the product about 2.1 instead of 2 per cent strength to insure that the bottled acid may be slightly above 2 per cent

immediately after bottling, and not much below 2 per cent before consumed in dispensing.

This preparation is frequently used in extremely critical cases and it must, therefore, be absolutely reliable. It should accordingly be tested from time to time and thrown away when no longer of proper strength.

Official Alternate Process.

The Pharmacopœia also gives an easily performed process for the extemporaneous preparation of diluted hydrocyanic acid, which is as follows:

Silver cyanide	6 parts
Hydrochloric acid	5 parts
Distilled water	55 parts

Mix these ingredients in a glass-stoppered bottle, adding the cyanide last, and shake well. When the precipitate has subsided, pour off the clear liquid, which will be the finished product, containing 2 per cent of HCy.

Reaction. $AgCy+HCl=AgCl+HCy$.

Another Method.

Diluted hydrocyanic acid may also be conveniently made from potassium cyanide with tartaric acid:

$$KCN+H_2C_4H_4O_6=KHC_4H_4O_6+HCN.$$

It will be seen from this equation that 65 parts of dry potassium cyanide and 150 parts of tartaric acid will furnish 27 parts of HCN. Hence the following formula may be used:

Potassium cyanide	22 parts
Tartaric acid	50 parts
Distilled water.	

Dissolve the cyanide and the tartaric acid, separately, each in 200 parts of distilled water. Add the solution of tartaric acid to the other, stirring well. Filter out the precipitated cream of tartar. Assay the filtrate and add enough distilled water to make the product contain exactly 2 per cent of HCN.

Theoretically the quantity of product obtained from these proportions should be 450 parts.

The diluted hydrocyanic acid prepared in this way may contain in solution a minute amount of tartaric acid and of cream of tartar.

Description.—Diluted hydrocyanic acid is a colorless liquid of a strong and characteristic odor and taste, resembling those of bitter almonds wetted with water, or of volatile oil of bitter almond. But as it is **extremely poisonous** it is dangerous to taste or to smell it without first diluting it considerably.

Preservation. Diluted hydrocyanic acid frequently undergoes some change by which a dark colored deposit is formed in it. According to Squibb, it may turn "nearly as black as dilute ink" within a year and a half, *if kept in glass-stoppered bottles,* and this change does *not* occur when the acid is put up in corked vials. The action which the acid thus appears to have upon the ground surface of glass has not been explained.

The preparation should always be kept in a cool, dark place.

DILUTED HYPOPHOSPHOROUS ACID.

ACIDUM HYPOPHOSPHOROSUM DILUTUM.

HPO_2H_2=66.

Calcium hypophosphite	17 parts
Oxalic acid	13 parts

Dissolve the hypophosphite in 100 parts of distilled water, and the oxalic acid in 50 parts. Filter both solutions. Add the solution of oxalic acid to the solution of calcium hypophosphite, stirring well. Filter. Wash the calcium oxalate on the filter with distilled water and mix the washings with the filtrate. Evaporate the liquid to 132 parts.

Reaction.

$$Ca(PO_2H_2)_2+H_2C_2O_4.2H_2O$$

$$=CaC_2O_4+2HPO_2H_2+2H_2O.$$

Notes. The product contains 10 per cent of HPO_2H_2. It must be kept in a glass-stoppered bottle.

LACTIC ACID.

ACIDUM LACTICUM.

$HC_3H_5O_3$=90.

Milk-sugar yields, by fermentation, lactic acid:

$$C_{12}H_{22}O_{11}+H_2O=4HC_3H_5O_3.$$

Several other organic substances, as dextrin, glucose, etc., are also capable of undergoing lactic fermentation.

When a mixture of 100 parts of sugar dissolved in sufficient water to yield a solution of about 1.06 sp. gr., with 8 to 10 parts of old cheese, and 50 parts of prepared chalk, is exposed for several weeks to a moderate heat, such as is afforded in a sunny place in the summer, calcium lactate is formed which is found crystallized in the liquid. The lactate of calcium is recrystallized, if necessary, and decomposed by sulphuric acid, yielding lactic acid and calcium sulphate.

Lactic acid is also prepared from milk-sugar and skimmed milk, a mixture of 300 Gm of milk-sugar and 4 liters of milk being exposed to a temperature of 20° to 30° C., and the acid neutralized from time to time with sodium bicarbonate, until the lactic fermentation ceases. The solution of sodium lactate is then evaporated to a syrupy consistence, dissolved in alcohol, and decomposed by sulphuric acid, after which the lactic acid is neutralized with chalk, and the lactate of calcium in turn decomposed to obtain a purer product. (See also, Ferrous Lactate).

Description.—The lactic acid of the American Pharmacopœia contains 75 per cent of absolute lactic acid ($HC_3H_5O_3$) and 25 per cent of water. It is a colorless, syrupy liquid, without odor, and of a pure acid taste. Absorbs water on exposure to moist air. Sp. w. about 1.213. Miscible with water, alcohol and ether in all proportions.

Valuation. To neutralize 4.5 Gm of lactic acid 75%

strength) requires 37.5 ml of normal solution of KOH. The indicator used is phenolphtalein.

NITRIC ACID.

ACIDUM NITRICUM.

HNO_3=63.

To obtain hydrogen nitrate, potassium or sodium nitrate is decomposed by hydrogen sulphate:

$$KNO_3+H_2SO_4=KHSO_4+HNO_3,$$
or
$$2NaNO_3+H_2SO_4=Na_2SO_4+2HNO_3.$$

Theoretically, 101 parts of potassium nitrate yield 63 parts of HNO_3, corresponding to about 92 parts of the official nitric acid (68 per cent HNO_3). The potassium-hydrogen sulphate does not decompose potassium nitrate until the temperature is so high as to decompose the nitric acid; hence it requires twice as much sulphuric acid to make nitric acid from potassium nitrate as it does to make the same quantity of nitric acid from sodium nitrate, which is decomposed by sodium-hydrogen sulphate. The proportions to be used are, therefore, 1010 parts of potassium nitrate and 980 parts of H_2SO_4 (corresponding to 1020 parts of 96 per cent sulphuric acid); or 850 parts of sodium nitrate and 510 parts of concentrated sulphuric acid.

If sodium nitrate be used, the sulphuric acid must be diluted with one-fourth its weight of water to prevent foaming. This foaming is caused by the abstraction of water from the nitric acid by the sodium-hydrogen sulphate, which results in the decomposition of the acid.

By using enough sulphuric acid to obtain a residue of acid sulphate, the reaction can be effected at a lower temperature than if the minimum quantity be employed, and there is then less decomposition of the nitric acid. The usual practice is to employ about 150 parts of concentrated sulphuric acid, diluted with 38 parts of water, to 170 parts of sodium nitrate.

The ordinary "half-gallon acid bottle" holds about 7 pounds of strong nitric acid.

Purification. The most common impurities in commercial nitric acid are H_2SO_4, Cl, N_2O_4, and iron nitrate. The chlorine is derived from the chlorides in the nitrates employed. Chili saltpeter also contains sodium iodide, which gives rise to contamination with iodine chloride. The iron comes from the iron cylinders in which the distillation is conducted in the manufacture of nitric acid on a large scale. Absolute nitric acid (1.52 sp. gr.) does not attack metallic iron, and the strong acid generated in the iron cylinders attacks the metal but little. Hence iron cylinders can be used. The yellow or reddish color of crude strong nitric acid is due mostly to nitrogen tetroxide, N_2O_4. This impurity may be removed by heating the acid to about 80° to 90° C., whereby it is driven off.

The purification of nitric acid is effected on a large scale by re-distillation from glass retorts. The first portion distilling over contains the nitrogen oxide, chlorine and iodine. As soon as the distillate is colorless and free from chlorine, the receiver is changed, and the distillation continued until about one-eighth remains. To fix the sulphuric acid in the crude nitric acid, a little potassium nitrate is added before the distillation.

Pure nitric acid is made directly from pure potassium nitrate and pure sulphuric acid.

Description.—A colorless, fuming liquid, of suffocating odor; extremely corrosive. Sp. w. about 1.414 at 15°. Contains 68 per cent of HNO_3.

The acid which manufacturers designate as "C. P." usually satisfies the pharmacopœial requirements.

Valuation. To neutralize 3.145 Gm of the official nitric acid requires 34 ml of normal solution of potassium hydroxide. Each ml of this volumetric solution corresponds to 2 per cent of absolute HNO_3. Phenolphtalein is the indicator used.

One ml of normal potassium hydroxide solution is the equivalent of 0.06289 Gm of HNO_3.

Action on metals. Gold, platinum, iridium, rhodium, and chromium are not affected by nitric acid. Iron, lead and silver are scarcely attacked by concentrated nitric acid, but diluted nitric acid dissolves them. Antimony, tin and tungsten are oxidized but not dissolved. Mercury, silver and bismuth dissolve readily,

especially in warm nitric acid, with the evolution of nitric oxide (NO) which, in contact with the air, at once oxidizes to nitrogen tetroxide (N_2O_4), which appears as red fumes.

Diluted Nitric Acid.

Nitric acid	100 Gm
Distilled water	580 Gm

Mix them. Keep the product in dark amber-colored, glass-stoppered bottles.

Diluted nitric acid contains 10 per cent, by weight, of absolute nitric acid.

Specific gravity: about 1.057 at 15° C.

To neutralize 6.29 Gm of diluted nitric acid should require 10 ml of normal solution of potassium hydroxide (each ml corresponding to 1 per cent of absolute acid), phenolphtalein being used as indicator.

NITROHYDROCHLORIC ACID.

ACIDUM NITROHYDROCHLORICUM.

Nitric acid	18 ml
Hydrochloric acid	82 ml

Mix the acids in a large glass beaker, and, when effesvescence has ceased, pour the mixture into a glass-stoppered bottle, which should not be more than half filled. Keep it in a cool, dark place.

Reaction. $NHO_3+3HCl=NOCl+2H_2O+2Cl$.

Notes. When strong nitric and hydrochloric acids are mixed both decompose, and free chlorine is formed in the liquid, which, therefore, is capable of dissolving gold, and has long been known under the name of "aqua regia." The medicinal value of the preparation also depends upon the free chlorine (and nitrosyl chloride) it contains.

The effervescence which always takes place should be allowed to cease before the product is bottled, the acids should be cold when mixed, and the finished preparation should be kept *in a cool place.*

The pharmacopœial direction to keep it in bottles not more than half filled is intended to prevent the possible bursting of the bottle should the evolution of gas not have been completed before it was bottled.

Care must be exercised in handling this acid. Chemical action must be completed before it, or any mixture containing it, should be dispensed. When mixtures of nitrohydrochloric acid with organic substances have been made explosions have happened a short time after they had been bottled.

Description.—A golden-yellow fuming and extremely corrosive liquid, having a strong odor of chlorine and a strongly acid reaction. Readily dissolves gold leaf.

Diluted Nitrohydrochloric Acid.

Nitric acid	40 ml
Hydrochloric acid	180 ml
Distilled water	780 ml

Mix the acids in a capacious glass vessel, and, when effervescence has ceased, add the distilled water. Keep the product in dark amber-colored, glass-stoppered bottles, in a cool place.

Description.—A colorless or pale yellowish liquid, having a faint odor of chlorine, and a very acid taste.

OLEIC ACID.

ACIDUM OLEICUM.

$HC_{18}H_{33}O_2$=282.

Impure oleic acid, known as "red oil," is obtained as a by-product in the manufacture of candles. Fats consist of olein, palmitin and stearin, which are the glycerides of oleic, palmitic and stearic acids.

The solid fatty acids (palmitic and stearic acids) are separated for use in making candles, and the residue, called "red oil," is crude oleic acid, which still contains some of the solid acids named.

This red oil is purified by exposing it to a temperature of about 4°.5 C. when the stearic and palmitic acids contained in it solidify and are removed by pressing and straining, after which the crude acid is shaken with sulphurous acid, washed, and filtered.

The product is far from being chemically pure, but is sufficiently pure for pharmaceutical and medicinal uses.

Description.—A pale yellow, or light sherry-colored, nearly inodorous and tasteless, oily liquid, of neutral or but faintly acid reaction. Exposed to the air it darkens, becoming brown, and assumes a decidedly acid reaction. Sp. gr. 0.860 to 0.890. Insoluble in water but readily soluble in alcohol, chloroform and ether.

OXALIC ACID.

ACIDUM OXALICUM.

$H_2C_2O_4.2H_2O=126$.

Oxalic acid occurs in many plants in the form of oxalates of calcium and potassium. It is, however, manufactured by heating sugar, starch, gum, saw-dust, or other organic matters with nitric acid, or with alkali hydrates. The most common process of manufacture consists in heating a pasty mixture of saw-dust and solution of soda and potassa at 204°.5 C. for an hour or two. The dried mass contains 28 to 30 per cent of oxalic acid as oxalates; by washing with sodium carbonate solution, the potassium oxalate is decomposed, leaving all of the oxalic acid in the form of sodium oxalate, the potassium carbonate being removed. The sodium oxalate is then treated with calcium hydrate, whereby calcium oxalate and sodium hydrate are produced. The calcium oxalate is then decomposed by sulphuric acid, and the oxalic acid purified by re-crystallization. The yield of oxalic acid equals about one-half of the weight of the saw-dust used.

Description.—Colorless or white prisms, readily soluble in water and alcohol. It has a strong acid taste, and is a powerful poison in large doses because so corrosive.

Pure oxalic acid volatilizes on platinum without residue.

Purified Oxalic Acid.

Commercial oxalic acid.....	1000 parts
Hydrochloric acid (31.9%HCl).....	300 parts
Water.	

Put the oxalic acid in a porcelain dish and add 700 parts of hot water. Boil for a few minutes. Let it settle. Decant the solution while still hot from the undissolved portion (which consists mostly of oxalates and may be reserved for further treatment, if profitable). Add the hydrochloric acid to the hot solution obtained. Stir well. Cool rapidly, stirring the liquid to get small crystals. Collect the crystals and wash the product with a very small quantity of cold water.

Recrystallize the product once or twice as may be required.

Notes. The less soluble oxalates remain undissolved when commercial oxalic acid is treated with less water than is required for complete solution. The remaining oxalates are decomposed by the hydrochloric acid, which is used in considerable excess. It is best not to allow the oxalic acid to form large crystals from the solution containing hydrochloric acid; small crystals are more readily washed free from the HCl. Recrystallization from a hot saturated solution is necessary to complete the purification, and it may be found necessary to repeat it.

Pure oxalic acid volatilizes on platinum without residue.

PHOSPHORIC ACID.

ACIDUM PHOSPHORICUM.

$H_3PO_4=98$.

Phosphoric acid can be made in various ways. It is most commonly made from phosphorus by oxidation with nitric acid. When strong nitric acid is used the reaction is more rapid and less heat is then applied; when the acid is diluted, the action being less violent, more heat is requisite.

It requires 26.9 parts of phosphorus to make 100 parts of the official phosphoric acid. The usual process is as follows:

Mix 510 Gm of nitric acid with 500 ml of distilled water in a tubulated retort capable of holding 2,000 ml. Place the retort

upon a sand-bath, or on a wire gauze support, connect it loosely with a well cooled receiver. Add 80 parts of phosphorus. Insert a funnel through the tubulus of the retort and then gradually apply heat until the reaction begins. Regulate the heat so as to prevent the action from becoming violent, checking it, if need be, by the addition of a little distilled water. Continue the digestion until all the phosphorus is dissolved, and return to the retort, from time to time, any liquid which may collect in the receiver.

When all the phosphorus is dissolved transfer the contents of the retort to a tared porcelain dish, and heat the liquid at a temperature of nearly but not above 190°C. until the excess of nitric acid is expelled and an odorless, syrupy liquid remains. Cool the dish and contents and add enough distilled water to make the total weight of the product 285 Gm.

Test the product for nitric, phosphorous and arsenic acids, all of which must be removed if present.

If nitric acid be present, evaporate the liquid until it ceases to give any further reaction for that acid; then let it cool and dilute it with distilled water until the prescribed weight (285 Gm) is restored.

If phosphorous acid be found in the phosphoric acid, add 30 Gm of nitric acid and again heat the mixture until reactions for phosphorous and nitric acids cease, and restore the prescribed weight (285 Gm) by dilution with distilled water as before.

Should arsenic be present, dilute the phosphoric acid with 750 ml of distilled water, heat the liquid to about 70° C. and then pass through it a stream of hydrogen sulphide gas for half an hour. Then remove the heat, but continue passing hydrogen sulphide through the dilute phosphoric acid until it becomes cold. Close the vessel tightly, set it aside for 24 hours, filter the liquid, then heat the filtrate until all odor of hydrogen sulphide has been expelled, filter again, and finally evaporate the now purified phosphoric acid until its weight is reduced to 285 Gm.

The product must be tested as to its strength in the manner prescribed below, and diluted or concentrated, as may be required.

Preserve the product in glass-stoppered bottles.

Reactions. Probably first—

$$2P+2HNO_3+H_2O=2H_3PO_3+NO.$$

Then

$$3H_3PO_3+2HNO_3=3H_3PO_4+H_2O+2NO.$$

Notes. The phosphorus used must be free from sulphur; otherwise sulphuric acid will be formed from the latter and contaminate the product. This result might, however, be prevented by using an excess of phosphorus so that some of it remains undissolved, the phosphorous acid being oxidized to phosphoric acid by a fresh portion of nitric acid after removing the undissolved phosphorus and sulphur.

As the phosphorus fuses collecting on the bottom of the retort, or flask, where it presents but a limited surface of contact for the action of the nitric acid, the oxidation is slow. High heat renders the action too violent, unless the nitric acid has been properly diluted, as in the usual process described above. Nitric acid of 1.20 sp. gr. is considered most suitable. Whenever the reaction is from any cause too violent, there is danger that particles of phosphorus may be thrown to the surface of the liquid and ignited, the heat from the combustion fracturing the vessel and scattering the burning phosphorus, the acid liquid, and the red nitrous fumes. The temperature need not exceed 50° C.

A flask can be used in place of the retort, and the heating can be done in a water-bath. In that case the neck of the flask should be covered by a watch crystal, the convex side downward, so that the liquid formed by the condensation of the rising vapors may run down into the flask again. Should the flask or retort break from any cause while undissolved phosphorus remains in it, the use of the water-bath instead of a sand-bath would prevent the ignition of the phosphorus.

Should the liquid, after the phosphorus has been dissolved, contain both phosphorous and arsenic acids, and should it then be concentrated to less than three times the weight of the phosphorus consumed, the phosphorous acid may be oxidized by the concentrated nitric acid so violently that the liquid froths over the sides of the dish unless a little water is added. The arsenic then separates as a brown powder of reduced metal.

If the liquid contains phosphorous acid, but no nitric acid, the phosphorous acid may be lost during the subsequent evaporation, hydrogen phosphide being formed unless nitric acid be added to prevent it.

$$4H_3PO_3=H_3P+3H_3PO_4.$$

The expulsion of the excess of nitric acid may be known to have been accomplished when a piece of filter-paper dipped in ammonia water no longer gives off white fumes when held over the liquid.

All of the nitric acid must be expelled before the liquid is charged with hydrogen sulphide gas for the purpose of removing arsenic present. Otherwise sulphur deposits, which is afterwards oxidized into sulphuric acid.

To facilitate the removal of the nitric acid, Professor Lloyd employs a little purified alcohol, and Professor Markoe adds a small amount of pure oxalic acid.

The evaporation of the acid must be done in well glazed porcelain dishes to avoid contamination with silicic acid. When silicic acid is present in considerable quantity in concentrated phosphoric acid, it separates in gelatinous flocculi upon dilution after standing some time.

Other methods. Phosphoric acid has also been made from phosphorus by the use of iodine or bromine and water. When bromine is employed the phosphorus combines with it, forming PBr_3 and PBr_5; the PBr_3 with $3H_2O$ yields H_3PO_3+3HBr, while the PBr_5 with $4H_2O$ yields H_3PO_4+5HBr; the H_3PO_3 with 2Br and H_2O will form H_3PO_4+2HBr. Thus ten molecules of HBr are formed simultaneously with two molecules of H_3PO_4, these acids being separated by distillation and both utilized. Analogous reactions take place when iodine or chlorine is used instead of bromine. These processes are, however, dangerous on account of the violent chemical action and the explosive character of the compounds of phosphorus with the halogens.

In other processes nitric acid is employed simultaneously with either bromine, or iodine, or both. The hydrogen acid formed is then decomposed by the nitric acid liberating the halogen, which again acts upon a new portion of the phosphorus:

$$3HBr+HNO_3=3Br+2H_2O+NO.$$

These processes are also dangerous. Markoe's method is probably the least dangerous of all of them. He mixed 540 parts of water, 90 parts of phosphorus, and 1 part of iodine in a stone

jar placed in ice-water; then 6 parts of bromine dissolved in dilute hydrobromic acid is very cautiously added. After the reaction 540 parts of nitric acid is added. After 24 hours the phosphorus is nearly or quite dissolved; if any of it remains undissolved after that period, heat may be safely applied to hasten its oxidation. The iodine, bromine and excess of nitric acid are then driven off by heat.

The slow oxidation of phosphorus by moist air at a low temperature has also been practiced.

Ortho-phosphoric acid has also been prepared by heating a solution of glacial phosphoric acid (metaphosphoric acid) in water, the reactions being: $2HPO_3+H_2O=H_4P_2O_7$ (pyrophosphoric acid), and then $H_4P_2O_7+H_2O=2H_3PO_4$.

A considerable quantity of pyrophosphoric acid may, however, remain undecomposed.

Amorphous phosphorus is readily oxidized by nitric acid without danger of fire.

The reaction begins after a few hours, generally without the application of any heat, and having once commenced it progresses very rapidly. If a capacious vessel is employed, no danger need be apprehended.

Properties. An aqueous solution containing 85 per cent of ortho-phosphoric acid and 50 per cent of water. It is a clear, colorless liquid, without odor, of a strongly acid taste and reaction. Sp. w. not less than 1.710 at 15°. Its sp. vol. is 0.5848.

Valuation. To neutralize 0.98 Gm of official phosphoric acid (85%) should require 17 ml of normal solution of potassium hydroxide. Each ml of the volumetric test solution corresponds to 5 per cent of H_3PO_4. Phenolphtalein is used as the indicator. The phosphoric acid should be somewhat diluted with water before the potassium hydroxide solution is added.

One ml of normal solution of potassium hydroxide is the equivalent of 0.0489 Gm of H_3PO_4 when forming K_2HPO_4 (phenolphtalein being then used as an indicator); it is equivalent to 0.0978 of H_3PO_4 when forming KH_2PO_4 (methyl-orange being the indicator used in this test).

[K_3PO_4 shows an alkaline reaction with all color indicators; K_2HPO_4 is neutral to phenolphtalein but alkaline toward methyl-orange and Congo-red; KH_2PO_4 gives an acid indicator with phenolphtalein but a neutral reaction with methyl-orange.]

Diluted Phosphoric Acid.

Phosphoric acid	100 Gm
Distilled water	750 Gm

Mix them. Keep the product in well-stoppered bottles.

Diluted phosphoric acid contains 10 per cent, by weight, of absolute H_3PO_4.

Specific gravity: about 1.057 at 15° C.

4.89 Gm of diluted phosphoric acid should require for neutralization 10 ml of normal potassium hydroxide solution (each ml corresponding to 1 per cent of the absolute acid), phenolphtalein being used as indicator.

METAPHOSPHORIC ACID.

HPO_3=80.

[Glacial phosphoric acid.]

This ice-like solid is formed by heating the ordinary phosphoric acid to redness, or by dissolving phosphorus pentoxide in cold water and heating. As found in commerce it usually contains much sodium phosphate.

SALICYLIC ACID.

ACIDUM SALICYLICUM.

$HC_7H_5O_3$=138.

Methyl salicylate exists in the volatile oil of gaultheria, which contains about 90 per cent of this compound ether. Salicylates have also been found in sweet birch and in other plants. Salicylic acid is manufactured as follows:

A solution of sodium hydroxide is saturated with phenol, the mixture evaporated to dryness with constant stirring, and the

residue, while still hot, is powdered; the powder is heated in a retort over an oil-bath to 100° C. whereby all moisture is driven off; then a current of dry carbonic acid gas is passed into the retort under slight pressure, the temperature being raised to 180° C., and finally to 220°—250° C. The residue now consists of sodium carbonate and sodium hydroxide together with sodium salicylate; it is dissolved in water, and hydrochloric acid is added, by which the salicylic acid is precipitated while sodium chloride remains in solution.

Reactions.

$C_6H_5OH+NaOH=C_6H_5ONa+H_2O$; then

$C_6H_5ONa+CO_2=NaC_7H_5O_3$; and

$NaC_7H_5O_3+HCl=HC_7H_5O_3+NaCl.$

Notes. Much depends upon a careful regulation of the temperature in this process. Potassium hydroxide cannot be used in place of the sodium hydroxide, the result being then para-oxybenzoic acid instead of salicylic acid. The chemical reactions include also the formation of "sodium-salicylate of sodium,"

$$NaC_7H_5O_3+C_6H_5ONa=NaC_7H_4(Na)O_3+C_6H_5OH;$$

this compound is decomposed by the HCl yielding salicylic acid and sodium chloride.

The crude salicylic acid is purified and decolorized by filtration of its solution through animal charcoal, and repeated recrystallization. To perfectly remove the resinous impurities, dialysation is resorted to.

When oil of wintergreen is treated with sodium hydroxide and the methyl salicylate of sodium boiled, methyl alcohol and sodium salicylate are formed.

Precipitated salicylic acid (amorphous) is not sufficiently pure for medicinal uses. The crystallized acid is required. Sublimed salicylic acid often has the odor of phenol, and acquires a reddish tint on exposure to air or sun light. The purest acid is that purified by dialysis.

Description.—Fine, white, light, prismatic, needle-shaped crystals, permanent in the air, free from odor of carbolic acid, but

sometimes having a slight aromatic odor, of a sweetish and slightly acrid taste and an acid reaction. Soluble in 450 parts of water and in 2.4 parts of alcohol at 15° C.; in 14 parts of boiling water; very soluble in boiling alcohol; also soluble in 2 parts of ether, in 2 parts of absolute alcohol; in 3.5 parts of amylic alcohol, and in 80 parts of chloroform. When heated to about 156° C. the crystals melt; at a higher temperature they are volatilized and decomposed.

SULPHURIC ACID.

ACIDUM SULPHURICUM.

$H_2SO_4=98$.

Crude or commercial sulphuric acid, still often called "oil of vitriol," is prepared from sulphur dioxide (obtained by burning sulphur or iron pyrites) by means of nitric acid in the presence of air and moisture in lead chambers. The

Reactions are as follows:

$2SO_2+2HNO_3+H_2O=2H_2SO_4+N_2O_3$; then

$N_2O_3+2SO_2+H_2O+O_2=2SO_2OHNO_2$; then

$2SO_2OHNO_2+H_2O=2H_2SO_4+N_2O_3$.

As sulphur and iron pyrites contain arsenic the crude acid formed in the lead chambers is contaminated with arsenic lead and nitric acid. The lead sulphate contained in the acid precipitates when the strong acid is diluted with more than three times its weight of water.

A much purer acid is necessary for pharmaceutical purposes. A process of purification is as follows:

A mixture of 300 parts of crude commercial sulphuric acid and 1 part of ammonium sulphate is distilled from a retort by gradually increased heat over the naked fire. The mixture is slowly raised to the boiling point, and the first 15 parts, containing chiefly water and sulphurous acid, are rejected, after which the next 225 parts are collected.

The object of adding the ammonium sulphate is to remove nitrogenous compounds by decomposition.

Notes. The pharmacopœias contain sulphuric acids varying in strength from 92.50 per cent to 98 per cent. That of the United States Pharmacopœia is required to contain *at least* 92.5 per cent of absolute acid, and in all official formulas wherever sulphuric acid is ordered, the quantity prescribed is based upon the assumption that the acid has that strength.

Sulphuric acid is extremely corrosive and destructive. It must therefore be handled with great caution. It reacts violently with all volatile oils and with a number of other substances. When it is mixed with water or with alcohol the chemical action which takes place causes a rapid and great rise of the temperature. Hence the only safe way to make these mixtures is to add the acid very slowly and in a small stream to the water or the alcohol, stirring constantly. It is also recommended that a very large vessel be used—one holding at least three times as much as the total quantity of mixture to be made, for if the liquid should become so hot as to boil or spurt damage might otherwise result.

Whenever it may be necessary to add fuming sulphuric acid ("Nordhausen acid"), or nearly absolute sulphuric acid to a weaker sulphuric acid in order to produce a mixture of definite strength, as, for instance, to make an acid containing exactly 98 per cent of absolute hydrogen sulphate, the stronger acid should be added to the weaker. And when a strong sulphuric acid is to be diluted with a comparatively small amount of water, the order of mixing should be as stated before—the acid must be added to the water and not the water to the acid.

The acid sold by manufacturers and dealers under the designation "chemically pure" (C. P.) is generally sufficiently pure for pharmaceutical purposes, fulfilling the requirements of the Pharmacopœia.

Sulphuric acid is customarily put up in bottles containing one, or two, or nine pounds. The ordinary so-called "five pint acid" bottle holds nine pounds of concentrated sulphuric acid.

Description.—A colorless, oily, odorless, extremely corrosive liquid. Sp. w. not less than 1.835 at 15°.

Action on metals. Concentrated sulphuric acid attacks copper, and, if hot, it also attacks mercury, silver and bismuth. Diluted sulpuhric acid attacks and dissolves iron, zinc, and nickel, but

does not dissolve platinum, gold, silver, lead, copper, mercury, aluminum, arsenic, antimony and bismuth.

Diluted Sulphuric Acid.

Sulphuric acid (92.5%).....	100 parts
Distilled water.....	825 parts

Pour the acid slowly, in a small stream, into the distilled water, under constant stirring.

Keep the liquid in glass-stoppered bottles.

Notes. When sulphuric acid and water are mixed a considerable rise of the temperature and a contraction of volume take place. If the liquids are mixed too rapidly, or the water added to the acid instead of *vice versa,* the heat generated may be so great as to cause serious accidents by violent boiling or spattering.

The diluted sulphuric acid obtained by the foregoing formula contains 10 per cent of absolute H_2SO_4.

Fuming Sulphuric Acid, or *Nordhausen Sulphuric Acid.*—This is a mixture of SO_3 and H_2SO_4, or possibly $H_2S_2O_7$. It is also called "pyrosulphuric acid" and "disulphuric acid." It is made by distilling ferrous sulphate which has been first dried to a certain point:

$$4FeSO_4+H_2O=2Fe_2O_3+H_2S_2O_7+2SO_2.$$

Fuming sulphuric acid is a thick liquid, extremely corrosive, and gives off dense fumes.

Haller's Acid Drops.

LIQUOR ACIDUS HALLERI.

This is a mixture of equal parts by weight of alcohol and sulphuric acid. The acid must be added to the alcohol gradually and cautiously, with constant stirring, in order that the temperature may not rise so high as to cause the liquid to boil. The liquid contains alcohol, sulphuric acid, ether, and ethyl-sulphuric acid. If the alcohol was pure, the preparation is colorless.

AROMATIC SULPHURIC ACID.

ACIDUM SULPHURICUM AROMATICUM—U. S.

Sulphuric acid (92.5%)	100 ml
Tincture of ginger	50 ml
Oil of cinnamon	1 ml
Alcohol, sufficient.	

Add the acid slowly and in small stream to 800 ml of alcohol, stirring constantly, being careful not to add the acid so rapidly as to cause the mixture to boil. When the mixture has cooled add the tincture and the volatile oil. Finally add enough alcohol to make the product measure 1,000 milliliters.

Keep it in glass-stoppered bottles.

Notes. Aromatic sulphuric acid contains about 20 per cent of sulphuric acid, chiefly in the form of ethyl-sulphuric acid or acid ethyl sulphate. It also contains some ether and various other products formed by the chemical action of the sulphuric acid on the other ingredients.

Description.—It is a pale yellowish-brown liquid of aromatic and strongly acid taste. Becomes darker by age through the carbonization of the organic matter by the sulphuric acid.

SULPHUROUS ACID.

ACIDUM SULPHUROSUM.

The "sulphurous acid" of the Pharmacopœia of the United States is an aqueous solution, representing 8.2 per cent of H_2SO_3, corresponding to 6.4 per cent of sulphur dioxide (SO_2).

It is prepared as follows:

Sulphuric acid	80 ml
Charcoal, in coarse powder	20 Gm
Distilled water	1 liter
Sodium carbonate	10 Gm

Put the acid and the charcoal in a flask of about 500 ml capacity. Connect the flask by means of bent glass tubing and perforated rubber stoppers with a wide mouthed bottle of about

200 ml capacity, to be used as a wash-bottle and for that purpose one-third filled with water. The wash-bottle is to be fitted with a rubber stopper having three perforations—one for the glass tube connecting it with the flask used as a generator, another for a safety tube, and the third for the glass tube connecting the wash-bottle with the receiver, which should consist of a bottle of about 1.5 liters capacity and containing 1 liter of water. The tube between the generator and the wash-bottle, and also the safety-tube, should reach nearly to the bottom of the wash-bottle; the tube connecting the wash-bottle with the receiver should end just below the stopper of the wash-bottle above the surface of the wash water, and should dip about three centimeters below the surface of the distilled water which is put into the receiver. Connect the receiver in the same manner with the second wide-mouthed bottle of 50 ml capacity containing the sodium carbonate dissolved in 30 ml of water. The joints between the pieces of glass tubing should be of soft rubber. Having made all the joints tight, apply heat to the flask by means of a sand-bath, and continue the heating until the evolution of gas has nearly ceased, keeping the receiver at or below 10° C. (50° F.) by means of cold water or crushed ice. Then remove the receiver, shake the contents, and keep the product in dark, amber colored, glass-stoppered bottles, completely filled, in a cool and dark place.

Test of strength. If 2 Gm of the sulphurous acid be diluted with 25 ml of distilled water and a little test-solution of starch added, it should require not less than 40 ml of decinormal test-solution of iodine to produce a permanent blue color in the liquid, which would show the presence of 6.4 per cent of SO_2 (each ml of the iodine solution being required for each 0.16 per cent of SO_2 contained in the product).

Reaction. $4H_2SO_4+2C=4SO_2+2CO_2+4H_2O$.

Notes. As soon as the reaction ceases the wash-bottle must be at once disconnected from the generator; otherwise the vacuum produced in the latter will cause the liquid in the wash-bottle to be forced back into the flask, causing its fracture. The solution of sodium carbonate in the end-bottle is to absorb the gas which is not absorbed by the water in the receiver.

Sulphurous acid deteriorates rather rapidly by oxidation to sulphuric acid.

Description.—A colorless solution with strong characteristic odor of burning sulphur, and sharp acid taste and reaction. Its specific gravity is 1.035 at 15° C.

Another Method.

Sodium bisulphite,
Diluted sulphuric acid,
Distilled water, of each sufficient.

Introduce the sodium bisulphite in a suitable roomy flask provided with stopper, safety tube and delivery tube, and connected with a wash-bottle containing some distilled water and, in turn, connected with a receiving bottle containing cold distilled water, previously freed from air by boiling.

Add the diluted sulphuric acid, a little at a time, through the safety tube, being careful not to add the acid too rapidly so as to cause a too rapid evolution of the gas.

When the water in the receiving bottle has been saturated bottle the product.

Reaction. $2NaHSO_3+H_2SO_4=Na_2SO_4+2H_2O+2SO_2$.

Notes. This process is not as economical as the official method, but it is more convenient and may often be found preferable. Theoretically 103.9 Gm of sodium bisulphite yields 64 Gm of SO_2, or enough to saturate one liter of water; but allowance must be made for the SO_2 held in the wash-bottle and that remaining in the various parts of the apparatus so that much more than that quantity is consumed. The quantity of diluted sulphuric acid required is about five times the amount of sodium bisulphite.

The size of the vessels, the amount of water in wash-bottle and receiver, the temperature of the water in the receiver, and certain other details, are indicated in the official process already described. The end bottle containing sodium carbonate should not be omitted.

Two receiving bottles may be alternately used, until the water in both shall have been saturated. Each bottle can then be shaken occasionally.

TARTARIC ACID.

ACIDUM TARTARICUM.

$H_2C_4H_4O_6$=150.

Tartaric acid occurs in grapes and in other fruits, in the form of acid potassium tartrate, from which it is manufactured.

The following formula is based on Scheele's process:

Cream of tartar	900 Gm
Prepared chalk	250 Gm
Calcium chloride	270 Gm
Sulphuric acid	260 ml
Distilled water, sufficient.	

Boil the cream of tartar with 6,500 ml of water, and gradually add the chalk, stirring constantly. When effervescence has ceased, add the calcium chloride dissolved in 800 ml of water. When the calcium tartrate has subsided, decant the liquid, and wash the tartrate with water until tasteless. Pour the sulphuric acid, previously diluted with 1,200 ml of water, on the calcium tartrate, mix thoroughly, boil for half an hour with repeated stirring, and filter through muslin. Evaporate the filtrate at a low temperature until it acquires the specific gravity 1.21, let it cool, and then separate and reject the crystals of calcium sulphate formed. Again evaporate the clear liquid until a pellicle forms, and then set aside to cool and crystallize. Lastly, purify the product by re-crystallization.

Reaction. $2KHC_4H_4O_6+CaCO_3 = CaC_4H_4O_6+K_2C_4H_4O_6+H_2O+CO_2$, and $K_2C_4H_4O_6+CaCl_2=CaC_4H_4O_6+2KCl$; finally, $CaC_4H_4O_6+H_2SO_4=H_2C_4H_4O_6+CaSO_4$.

Notes. Tartaric acid crystallizes best from a solution containing a small amount of free sulphuric acid. Calcium hydroxide can be used instead of calcium carbonate.

Description.—Colorless prisms, having a sharp but pleasant acid taste. Readily soluble in water and alcohol, and slightly soluble in ether. The crystals are anhydrous and not deliquescent.

VALERIANIC ACID.

ACIDUM VALERIANICUM.

(*Acidum Valericum—Valeric Acid.*)

$HC_5H_9O_2=102.$

This acid exists in valerian, anthemis, angelica, humulus and viburnum, being formed by the oxidation of volatile oils or other natural constituents, and accordingly present in larger proportion in old lots of these plants which have been somewhat exposed to the air. It is now prepared by the action of chromic anhydride upon amyl alcohol, as illustrated in the process for the manufacture of sodium valerate. See "Sodium Valerate."

Valeric acid may be conveniently prepared by adding sulphric acid to a strong solution of sodium valerate in water. The oily layer rising to the surface of the mixture is valeric acid. This is separated from the watery liquid containing the sodium sulphate, then deprived of water by repeatedly shaking it with strong sulphuric acid, and, after separation from the sulphuric acid, the product is distilled.

Description.—A thin, oily, colorless liquid, having a very characteristic, sharp, disagreeable, penetrating, valerian-like odor. Its taste is pungent, acid, extremely disagreeable. Soluble in from 26 to 30 parts of water. Soluble in all proportions in alcohol and ether. Its sp. w. is about 0.9536 at 0° C.

OTHER PREPARATIONS.

ALUM.

ALUMEN.

(*Potassium Alum. Aluminum-Potassium Sulphate.*)

$AlK(SO_4)_2.12H_2O=474.$

Aluminum sulphate	25 parts
Potassium sulphate	7 parts
Water	60 parts
Diluted sulphuric acid (10%)	1 part

Heat the water to boiling. Add the aluminum sulphate. Then add the potassium sulphate, stirring well. Add the diluted sulphuric acid. Filter if necessary. Set the solution aside to cool and crystallize.

Evaporate the mother-liquid to obtain additional crops of crystals as far as practicable. Recrystallize the product once or twice as may be necessary.

Notes. The aluminum sulphate used may be that obtained from cryolite, as described under Aluminum Sulphate.

Crystallized Alum.

Potash alum	2 parts
Water	18 parts
Diluted sulphuric acid	1 part

Make a solution with the aid of heat. Filter. Crystallize the salt in the usual way.

Very large and well-developed crystals may be easily obtained if the crystallization proceeds slowly by spontaneous evaporation and with the solution at perfect rest. To start crystallization a few small crystals of pure potash alum may be placed in the solution. The crystals should not be permitted to rest on one side throughout the process, but should be turned over occasionally in order that they may be equally well developed on all sides.

Turbidated Alum.

Potash alum	20 parts
Boiling water	20 parts
Diluted sulphuric acid	1 part

Dissolve, filter, and cool the solution rapidly, stirring it constantly, to as low a temperature above the freezing point of water as may be conveniently attained. Collect, drain and dry the crystalline salt.

Description.—Alum consists of large, colorless, octohedral crystals, sometimes modified by cubes, or occurs in crystalline fragments, without odor, but having a sweetish and strongly astringent taste. On exposure to the air, the crystals are liable to absorb ammonia, and acquire a whitish coating.

Soluble in 9 parts of water at 15° C., and in 0.3 part of boiling water; it is also freely soluble in warm glycerin, but is insoluble in alcohol.

The salt has an acid reaction upon litmus paper.

Dried Alum.

ALUMEN EXSICCATUM.

$KAl(SO_4)_2$=258.

Expose 11 parts of crystallized potash alum for several days to a temperature of about 80° until it has thoroughly effloresced. Then heat it in a porcelain dish gradually up to 200°, being careful not to permit the temperature to rise above 205°, and continue the heating until a porous white mass remains, weighing 6 parts. When cold, powder it and keep it in a well-closed bottle.

Notes. Potassa alum contains 45.57 per cent of water of crystallization. When kept heated to 40° C. it loses about 2.7 per cent of that water; at 47° C. it loses 9.6 per cent; at 60° C. it loses most of its water, but the pieces still retain to a great extent their shape, and the product, which is not porous, yields a clear solution with water. At 80° C. the salt falls to powder, but still retains a considerable amount of water. Long-continued drying at 100° C.

expels all the rest of the water and leaves an entirely water soluble product, but not a light and porous one.

When first effloresced at 80° C. and then at once gradually to between 200° and 205° C. and kept at that temperature until its weight is reduced to that of the anhydrous product, the result is a light and porous dried alum.

When alum is at once heated to 92° C. it undergoes aqueous fusion, and if then set aside to cool the liquid does not solidify until after long standing.

A porous and light product is to be much preferred, and such a product can not be obtained if the alum be permitted to dissolve in its water of crystallization. When heated at a too low temperature the alum may form a glassy mass which can not be rendered porous. In this glassy condition the alum is said to retain 14 molecules of water which can be expelled at a higher temperature, but the product is then heavy and non-porous.

During the latter part of the process of heating the alum it swells and foams considerably from the water vapor formed. Hence the dish used should be large enough, or only about one-fourth to one-third filled. The alum should not be stirred during the heating; if stirred it will not yield a light product.

As it is impracticable for the student to regulate the temperature so as to maintain it at between 200° and 205°, he may not succeed in making a perfect product; but he can verify in a general way the statements here made and may even produce a better dried alum than is obtained by the official process (U.S.P., 1890), which is as follows:

"Place 100 grams of alum [in small pieces] in a shallow porcelain capsule so as to form a thin layer, and heat it on a sand-bath until it liquefies. Then continue the application of a moderate heat, with constant stirring, until aqueous vapor ceases to be disengaged, and a dry, white, porous mass is obtained, weighing 55 grains. When cold, reduce the product to fine powder."

Dried alum should be kept in tightly closed bottles. Exposed to a moist atmosphere it absorbs water quite rapidly, even to the extent of about 18 molecules, or over 50 per cent of its weight.

Description.—Porous white masses, or a white granular powder, odorless, sweetish, astringent. Slowly soluble in 20 parts of water at 15°.

AMMONIA ALUM.

ALUMINI ET AMMONII SULPHAS.

Aluminum-Ammonium Sulphate.

$AlH_4N(SO_4)_2.12H_2O=453.$

Aluminum sulphate	25 parts.
Ammonia water (10% of H_3N)	13 parts.
Diluted sulphuric acid (10%)	35 parts.
Water	10 parts.

Mix the diluted acid and water in a porcelain dish. Add the ammonia water gradually, stirring well. Then add the aluminum sulphate; heat to boiling for a few minutes; filter. Set aside to cool and crystallize.

Additional quantities of crystals may be obtained on evaporation of the mother-liquors.

Recrystallize the product once or twice to purify it.

Notes. The aluminum sulphate required may be obtained from cryolite by the process described under Alumini Sulphas.

Description.—Colorless, transparent, octohedral crystals; odorless; taste sweetish, astringent

Commercial "alum" is now "ammonia alum," while the official alum is "potash alum."

For most of the purposes for which alum is used, the ammonia alum is as effective as the potash alum. But "dried alum" or "burnt alum" must be made from potash alum, and when alum is prescribed for pharmaceutical or medicinal purposes the official "alumen" is, of course, the kind intended.

Ammonia alum is somewhat more readily soluble in water than the potash alum; but in other respects their physical properties are alike. On warming the ammonia alum with solution of potassium hydroxide the odor of ammonia is developed.

ALUMINUM ACETATE SOLUTION.

[After the German and Swiss Pharmacopœias.]

Aluminum sulphate	222 parts.
Acetic acid (96%)	300 parts.
Distilled water	480 parts.

Make a solution. Add to this, gradually, a mixture made of—

Calcium carbonate	100 parts.
Distilled water	140 parts.

Let the mixture stand 24 hours, stirring or shaking it occasionally.

Filter the liquid, and add, through the filter, enough distilled water to make the whole product weight 1000 parts.

Description.—A clear colorless liquid of 1.058 sp. w., containing 10 per cent of anhydrous basic aluminum acetate, corresponding to 3.5—3.6 per cent dry aluminum oxide. It has a faint odor of acetic acid, a sweetish astringent taste and an acid reaction.

Used as a disinfectant.

Aluminum Aceto-Tartrate Solution.

The Swiss and other pharmacopœias contain the following formula:

Solution of aluminum acetate	74 parts.
Tartaric acid	3 parts.
Distilled water	23 parts.

Dissolve the tartaric acid in the solution of aluminum acetate, and add the water.

Description.—A colorless solution of 1.047 sp. w. Yields upon evaporation 10 per cent of aluminum aceto-tartrate dried at 100° C. Reaction acid. A disinfectant.

ALUMINUM CHLORIDE.

ALUMINI CHLORIDUM.

$AlCl_3.6H_2O=241.2.$

Saturate diluted hydrochloric acid with aluminum hydroxide, prepared as described under that head. Acidulate the solution by adding some more of the acid; filter and evaporate to crystallization over a water bath.

It decomposes when evaporated to dryness.

This preparation may also be made by metathesis from aluminum sulphate and barium chloride.

Description.—Colorless crystals. Soluble in 0.25 part of water at 15°.

Used as a disinfectant and an astringent.

Aluminum Chloride Solution.

LIQUOR ALUMINI CHLORIDI.

Solution of aluminum chloride is often employed as a disinfectant. Pure aluminum chloride is not necessary for this purpose. A good product is obtained as follows:

Calcium chloride	1 part
Alum, in powder	2 parts
Water.	

Dissolve the calcium chloride in 5 parts of water and the alum in 15 parts of boiling water. Mix the solutions. Filter. Add enough water to make the whole product weigh 20 parts.

Notes. The product contains aluminum chloride, potassium chloride, and a trace of calcium sulphate.

ALUMINUM HYDROXIDE.

ALUMINI HYDROXIDUM.

$Al(OH)_3 = 78.$

Alum, in powder	1 part
Sodium carbonate	1 part
Distilled water, sufficient.	

Dissolve each salt in 15 parts of distilled water, filter the solutions and heat to boiling. Then having poured the hot solution of carbonate of sodium into a capacious vessel, gradually pour into it the hot solution of alum with constant stirring, and add about 10 parts of boiling distilled water. Let the precipitate subside, decant the clear liquid, and pour upon the precipitate 20 parts of hot distilled water. Again decant, transfer the precipitate to a strainer, and wash it with hot distilled water until the

washings give but a faint cloudiness with test-solution of barium chloride. Then allow it to drain, dry it at a temperature not exceeding 40° C., and reduce it to a uniform powder.

Reaction.

$$3Na_2CO_3+2AlK(SO_4)_2+3H_2O$$
$$=3Na_2SO_4+K_2SO_4+2Al(OH)_3+3CO_2.$$

Notes. It is necessary to have a decided excess of alkali present throughout the process, and as equal parts of alum and sodium carbonate leave only about ten per cent excess of the alkali, and as this excess is not too great, these proportions are used. Unless a sufficient excess of alkali be present from the beginning to the end of the reaction, and the liquid still remains alkaline after all the alum has been decomposed, the product will contain sulphate. This explains why it is directed that the alum solution must be poured into the alkali solution, and not *vice versa;* it also accounts for the injunction to add the alum solution slowly and with constant stirring.

Aluminum hydroxide is a very light, voluminous precipitate, forming an almost gelatinous magma, which settles very slowly if at all, and is therefore difficult to wash. It will of course settle less readily in a dense liquid than in one less dense. Hence the addition of hot water greatly facilitates the washing by decantation. But prolonged contact with boiling water changes the constitution of the hydroxide from $Al(OH)_3$ to OAlOH, and the latter compound is insoluble in acids. Hence, the liquid in which the hydroxide is suspended must not be boiling, but only hot.

The washed hydroxide is to be dried without much heat in order to avoid its becoming hard and gritty.

Aluminum hydroxide may also be prepared from cryolite, as described under the title of Aluminum Sulphate.

It is chiefly prepared as a preliminary step in the preparation of sulphate, chloride, nitrate, acetate, and other aluminum salts. When prepared for this purpose the hydroxide, after having been well washed, is simply drained but not dried before dissolved in the proper acid.

Description.—A white, light, amorphous, odorless and tasteless powder, insoluble in water and in alcohol.

ALUMINUM NITRATE SOLUTION.

LIQUOR ALUMINI NITRATIS.

Containing about 10 per cent of $Al(NO_3)_3 9H_2O = 375$.

Alum, powdered	33 parts
Sodium carbonate	30 parts
Nitric acid	20 parts

Dissolve the alum and the sodium carbonate separately, each in 450 parts of boiling water; filter the solutions; pour the solution of alum into the solution of sodium carbonate, stirring briskly. Add 300 parts of boiling water. Digest the mixture at a gentle heat until the evolution of carbon dioxide ceases; wash the precipitate with hot distilled water, first by decantation and afterward on a muslin strainer, until the washings cease to give a precipitate with test-solution of barium chloride. Let the magma drain thoroughly, and then dissolve it in the nitric acid. Evaporate the solution to 260 parts.

Notes. The reaction taking place between the alum and sodium carbonate, and notes on that part of the process, are given under the title, aluminum hydroxide.

This solution is employed as an astringent and disinfectant.

ALUMINUM OLEATE.

ALUMINI OLEAS.

$Al(C_{18}H_{33}O_2)_3 = 870$.

Potash alum	60 Gm
White Castile soap, in fine powder	110 Gm

Dissolve the alum in 3,600 ml of water, and the soap in 1,800 ml of hot water. Pour the cold soap solution gradually into the alum solution. Warm the mixture until the oleate separates,

Decant the mother liquor, wash the oleate twice with hot water, using 3,000 ml of water each time, and then collect the product.

Reaction.

$$6NaC_{18}H_{33}O_2+2KAl(SO_4)_2$$
$$=2Al(C_{18}H_{33}O_2)_3+K_2SO_4+3Na_2SO_4.$$

Notes. To free this oleate from water it must be melted in an evaporating dish by very gentle heat over the water-bath.

The yield is about 100 Gm or slightly over that amount.

Description.—Aluminum oleate is a soft, white plaster, containing 5.86 per cent of aluminum oxide.

ALUMINUM SULPHATE.

ALUMINI SULPHAS.

$$Al_2(SO_4)_3.16H_2O=630.$$

Powdered cryolite	10 parts
Lime in powder	8 parts
Sodium bicarbonate, about	12 parts
Diluted sulphuric acid, about	140 parts
Water.	

Slake the lime with 3 parts of water. Add 40 parts of water and mix well. Then add the powdered cryolite, stirring thoroughly. Boil for six or eight hours, stirring frequently, and replacing from time to time the water lost by evaporation. Filter the mixture through a muslin strainer, and wash the residue on the strainer with hot water, adding the washings to the previous filtrate. Boil the liquid, adding small quantities of sodium carbonate until all the iron has been precipitated (as may be determined by the addition of test-solution of potassium ferrocyanide to a test-portion acidified with hydrochloric acid).

Then add a solution of sodium bicarbonate to the hot liquid as long as precipitation is produced, taking care not to add an excess. Wash the precipitated aluminum hydroxide with hot water, until the washings are free from sodium carbonate. Dissolve the washed aluminum hydroxide in the diluted sulphuric acid, evaporate the solution to a density of about 1.20 and set it

aside to crystallize, or evaporate to a syrupy consistence, and set it aside to solidify on cooling.

Reaction.

$AlF_3.3NaF+3CaO=Al(NaO)_3+3CaFe_2$; then

$Al(NaO)_3+3NaHCO_3=Al(OH)_3+3Na_2CO_3$; and, finally,

$2Al(OH)_3+3H_2SO_4+10H_2O=Al_2(SO_4)_3.16H_2O$.

Notes. This aluminum sulphate is used for the preparation of alum (see alum and ammonia alum).

The quantity of sodium bicarbonate indicated may not all be required.

It is necessary that the diluted sulphuric acid should be completely saturated with aluminum hydroxide, or, in other words, that some of that hydroxide should remain undissolved in the acid. Hence it is recommended that only three-fourths of the diluted acid be used at first, and that after all of the aluminum hydroxide shall have been added, the remainder of the sulphuric acid be added gradually according to the indications. The whole quantity named can probably be saturated with the aluminum hydroxide obtained from the quantity of cryolite used unless a portion of the hydroxide be lost in the process of washing. But the results are somewhat variable and it is therefore necessary to proceed cautiously.

Pure Aluminum Sulphate.

$Al_2(SO_4)_3.16H_2O=630$.

Alum	30 parts
Sodium carbonate	30 parts
Sulphuric acid	9 parts

Dissolve the alum and the sodium carbonate, each in 400 parts of boiling water, filter, and pour the hot alum solution into the hot solution of sodium carbonate, stirring constantly. Digest the mixture at a gentle heat until the evolution of carbon dioxide ceases. Wash the precipitate with hot water until the washings no longer yield a precipitate with test solution of barium chloride. Then let the precipitated aluminum hydrate drain as far as possible, and dissolve it with the aid of gentle heat in the sulphuric

acid, previously diluted with 50 parts of water; filter the solution, evaporate it with constant stirring, over a water bath, until a nearly dry white salt remains.

Notes. Aluminic hydroxide is first prepared. See article Aluminum Hydroxide, p. 258, for reaction and notes. Then the sulphuric acid is saturated with the aluminum hydroxide. Care must be taken not to lose any of the hydroxide in the process of washing it, for if the quantity is insufficient the acid can not be saturated and the product will then contain free sulphuric acid. If there is any reason for believing that any portion of the aluminum hydroxide has been wasted, the amount of sulphuric acid used may be reduced so as to insure its saturation.

Description.—Pure aluminum sulphate is a white crystalline powder; odorless; taste sweetish, astringent. Permanent in the air if properly prepared. Soluble in 1.2 parts of water at 15°. Insoluble in alcohol.

AMMONIA WATER.

LIQUOR AMMONII HYDROXIDI.

H_4NOH=35, dissolved in water.

Ammonium chloride, in coarse powder..	80 parts
Lime	100 parts
Distilled water, water, each sufficient.	

Slake the lime with 2 parts of water; add the ammonium chloride to 20 parts of water; mix the whole well, and immediately introduce the mixture into an iron flask. Connect the flask at once, by means of corks, glass tubes and rubber stoppers, with a Woulff bottle of the capacity of 20 parts. Connect this with a second Woulff bottle of the same size, and this in turn with a receiver of the capacity of 160 parts, and containing 20 parts of distilled water. Connect the receiver with still another bottle containing 16 parts of distilled water. The Woulff bottles are to be empty. The second Woulff bottle and the receiver must each be provided with a siphon safety tube charged with a very short column of mercury.

The iron flask is placed in a sand-bath, and heat is applied. The receiver is to be cooled by cold water. All joints being tight, the heat is gradually raised and continued until no more gas passes over.

Reaction. $2H_4NCl+Ca(OH)_2=2H_4NOH+CaCl_2$.

Notes. For experimental purposes a glass flask may be used instead of that of iron, but the process cannot then be continued until all the gas has been obtained which the ammonium chloride will yield.

The water is added to the lime and ammonium chloride in order to facilitate the reaction. The calcium hydrate must be allowed to become cool before mixing it with the ammonium chloride.

At the conclusion of the operation there will be considerable quantities of liquid in all four bottles. The Woulff bottles will contain discolored ammonia solutions; the third bottle or receiver, contains a strong solution of ammonia, which is to be diluted with distilled water until of the required strength; the terminal bottle contains weaker ammonia solution, which may be strengthened by charging it with an additional quantity of gas obtained by heating in a flask the contents of the Woulff bottles.

If ammonium carbonate is used instead of chloride, less heat will be required, but in that case the gas must be passed through a bottle containing milk of lime to fix the carbon dioxide.

In order to prevent accidents which may cause serious results the Pharmacopœia directs that stronger ammonia water shall be kept in *strong* glass stoppered bottles, only partially filled, and put in a cool place. If the containers are not sufficiently strong, and are too nearly full and kept in a warm place, they may burst. The gas is extremely caustic and acrid. To inhale or smell ammonia without due caution is attended with danger, and a full container with tightly fitting glass-stopper should never be opened, especially if it is not cool, without turning the face aside from it.

Valuation. Two water-solutions of ammonium hydroxide are contained in the Pharmacopœia of the United States. One is called **aqua ammoniae, ammonia water,** and is required to be of a strength corresponding to 10 per cent of H_3N; the other is called

aqua ammoniae fortior, stronger water of ammonia, representing 28 per cent of H_3N.

To neutralize 3.4 Gm (or 3.54 ml) of ammonia water requires 20 ml of normal sulphuric acid; to neutralize 1.7 Gm (or 1.88 ml) of stronger ammonia water requires 28 ml of normal sulphuric acid. Each ml of normal sulphuric acid required to neutralize a solution of ammonium hydroxide corresponds to 0.017 Gm of H_3N. The indicator prescribed in the United States Pharmacopœia is rosolic acid.

Description.—Colorless, transparent, suffocatingly pungent, acrid, alkaline.

The ammonia water (10 per cent H_3N) has the sp. w. 0.960 at 15°; the stronger ammonia water (28 per cent H_3N) has the sp. w. 0.901 at the same temperature.

AMMONIA SPIRIT.

SPIRITUS AMMONIAE.

An alcoholic solution of ammonia containing 10 per cent of the gas.

Stronger water of ammonia.....	500 ml
Alcohol	1,000 ml

Put the ammonia water into a boiling-flask of the capacity of about 1,000 ml; connect this with a bottle of 1,500 ml capacity to be used as a receiver and containing the alcohol. The bent glass tube connecting the flask and the receiver should be of about 10 millimeters' diameter. Place the receiver in ice-cold water. Fit a thermometer in the flask.

Now heat the flask carefully and gradually, by means of a water bath, up to about 60° C., and maintain this temperature in the bath for ten minutes. Then disconnect the receiver and test the solution of ammonia with normal sulphuric acid to determine its ammoniacal strength; 20 ml of that acid should exactly neutralize 3.4 Gm or 4.2 ml of official spirit of ammonia. Having ascertained the quantity of normal sulphuric acid required to neutralize the product, adjust the strength accordingly to the required standard.

Notes. The solution obtained is usually stronger than 10 per cent, in which case it simply requires dilution with the proper proportion of alcohol. If the solution be found to contain less than 10 per cent of H_3N the receiver may be reconnected and the process continued. Should the stronger water of ammonia used fail from any cause to furnish sufficient H_3N to give the product the requisite strength, put a fresh portion of ammonia water in the flask and repeat the process.

It has the sp. gr. 0.810, and the sp. vol. 1.235.

Aromatic Spirit of Ammonia.

Ammonium carbonate, in translucent hard pieces	34 Gm
Ammonia water	90 ml
Oil of lemon	10 ml
Oil of lavender flowers	1 ml
Oil of nutmeg	1 ml
Alcohol	700 ml
Distilled water.	

To the ammonia water, contained in a flask, add 140 ml of distilled water, and afterwards the ammonium carbonate reduced to a moderately fine powder. Close the flask and agitate the contents until the carbonate is dissolved. Introduce the alcohol into a graduated bottle of suitable capacity, add the oils, then gradually add the solution of ammonium carbonate, and afterwards enough distilled water to make the product measure one liter. Set the liquid aside during twenty-four hours in a cool place, occasionally agitating, then filter it through paper, in a well-covered funnel.

Keep the product in glass-stoppered bottles, in a cool place.

Description.—A nearly colorless liquid when freshly prepared, but gradually acquiring a somewhat darker tint.

It has a pungent, ammoniacal odor and taste.

Specific gravity: about 0.905 at 15° C.

AMMONIUM ACETATE SOLUTION.

LIQUOR AMMONII ACETATIS.

[*Spiritus Mindereri.*]

An aqueous solution containing about 7 per cent of ammonium

acetate ($H_4NC_2H_3O_2$=77) together with small quantities of acetic acid and carbonic acid.

Ammonium carbonate	5 Gm
Diluted acetic acid	100 ml

Add the ammonium carbonate gradually to the cold diluted acid, and stir until it is dissolved.

This preparation should be freshly made when wanted.

Reaction.

$$H_4NHCO_3.H_4NNH_2CO_2+3HC_2H_3O_2$$
$$=3H_4NC_2H_3O_2+H_2O+2CO_2.$$

Notes. The ammonium carbonate used must be in hard translucent pieces, free from white pulverulent bicarbonate.

To make a preparation such as the Pharmacopœia intends, the diluted acid should be cold in order that the free carbonic acid which is formed may dissolve in sufficient quantity in the liquid. The presence of this carbonic acid renders it impracticable to determine the point of neutralization by means of test-paper; but the preparation, as made, is slightly acid. The solution should have a pure acidulous saline taste, or the saline taste modified only by that of the carbonic acid; a slight excess of acetic acid is desirable; but any excess of alkali is objectionable. Even when neutral the solution gives an acid reaction to litmus paper owing to the carbonic acid.

When the ammonium carbonate dissolves in the acetic acid the temperature is lowered, on account of the liberation of carbonic acid gas. This fall of the temperature of the liquid is advantageous, as it aids the solution of another portion of the gas.

In some pharmacopœias ammonia water is used instead of ammonium carbonate. In this case the temperature rises. Test-paper can be used to ascertain the reaction when this method is followed, but it is directed that the solution shall be, not neutral, but slightly acid.

The preparation must not be filtered as that would expel nearly all the carbonic acid.

AMMONIUM CITRATE SOLUTION.

LIQUOR AMMONII CITRATIS.

A solution of ammonium citrate each ml of which contains 0.50 Gm of $(H_4N)_3C_6H_5O_7$ is often employed in clearing certain liquids containing iron compounds and liable to become cloudy or to form precipitates. Small additions of such a solution to the preparations known as "beef, wine and iron" and elixirs containing phosphate or pyrophosphate of iron associated with organic substances prevent the formation of certain precipitates containing iron, or dissolve them when formed. Such a solution may also be advantageously employed in the preparation of soluble phosphate and pyrophosphate of iron.

It is prepared as follows:

Citric acid	500 Gm
Stronger ammonia water (28% of H_3N)	430 ml
Distilled water.	

Dissolve the acid in the ammonia water. Neutralize perfectly by adding as much ammonia water as may be required. Then add enough distilled water to make the whole measure 1,250 ml.

Solution of Di-ammonium-Hydrogen Citrate,

may be prepared by adding 250 Gm of citric acid to 1,250 ml of the solution of normal ammonium citrate just described and then diluting with enough distilled water to make the whole measure 1,800 ml.

Each ml of this solution contains 0.50 Gm of di-ammonium-hydrogen citrate $(H_4N)_2HC_6H_5O_7$.

This solution is frequently used in the preparation of the soluble phosphate and pyrophosphate of iron.

AMMONIUM BENZOATE.

AMMONII BENZOAS.

$H_4NC_7H_5O_2=139$.

Benzoic acid	20 parts
Distilled water	40 parts
Water of ammonia, sufficient.	

Dissolve the benzoic acid in 33 parts of water of ammonia previously diluted with the water. Evaporate over a water-bath by gentle heat, adding a little more ammonia water from time to time, if necessary to keep the alkali in constant excess, so as to prevent the formation of acid ammonium benzoate, which is less soluble. When reduced to 50 parts set aside to crystallize. Collect and dry the crystals. The mother liquor will yield more crystals after evaporation, keeping the ammonia slightly in excess as before.

Reaction. $HC_7H_5O_2+H_4NOH=H_4NC_7H_5O_2+H_2O$.

Description.—White, thin, lamellar crystals, odorless or having a faint odor of benzoic acid; soluble in 5 or 6 parts of cold water, 1.2 parts of boiling water, 30 parts of alcohol, 7.6 parts boiling alcohol, or 8 parts of glycerin.

Should be kept in tightly closed bottles.

AMMONIUM BICARBONATE.

AMMONII BICARBONAS.

$H_4NHCO_3=79$.

Stronger ammonia water (28% of H_3N), any desired quantity.

Carbon dioxide.

Conduct a stream of carbon dioxide into the stronger ammonia water contained in a flask keeping the flask and contents cool with running water. Continue the current of CO_2 until the gas is no longer absorbed. The salt crystallizes from the solution. Separate the crystals by turning the contents of the flask into a funnel provided with a strainer or perforated diaphragm; drain well, and dry them over sulphuric acid.

Keep the product in a tightly closed bottle in a cool place.

Reaction. $H_4NOH+CO_2=H_4NHCO_3$.

Notes. Erdmann recommends putting the concentrated ammonia water in a small flask closed by a stopper bearing a short glass tube extending down nearly to the surface of the liquid but not dipping into it. The carbon dioxide is to be conducted into the flask through that tube. Normal ammonium carbonate

is first precipitated but redissolves. After the liquid has stood for some time under the pressure of the gas, the bicarbonate is separated. When no more crystals are formed a stratum of alcohol cautiously laid over the mother liquor will cause the deposition of an additional crop. The salt may best be preserved in glass tubes filled with carbon dioxide and sealed.

Another Method.

Dissolve out the ammonium carbonate from powdered ordinary ammonium carbonate by macerating it with twice its weight of alcohol for a few minutes. After separating the alcoholic solution of the carbonate, wash the undissolved ammonium bicarbonate with another portion of alcohol, using the same quantity as before. Expose the product to the air until the alcohol has evaporated, and then bottle it.

The carbonate may also be removed by macerating the ordinary ammonium carbonate for two hours with twice its weight of water.

Description.—Colorless crystals, or a white crystalline powder of ammoniacal taste and faintly ammoniacal odor (or nearly odorless if quite dry). Soluble in 5.5 parts of water at 15°. Insoluble in alcohol.

AMMONIUM BROMIDE.

AMMONII BROMIDUM.

$H_4NBr=98$.

Iron wire	4 parts
Bromine	12 parts
Ammonia water	25 parts
Distilled water.	

Put the iron wire into a flask, add 50 parts of warm (not *hot*) water, and then gradually add 9 parts of bromine. Put a loose plug of cotton into the neck of the flask, and shake gently from time to time until a greenish liquid is obtained having no odor of bromine. Filter this solution of ferrous bromide, add the remainder of the bromine to the filtrate and shake gently so that a uniform solution may be obtained.

Pour this solution into the ammonia water previously diluted with 50 parts of distilled water. Shake well. Heat the mixture over the water-bath for half an hour. Then filter. When the liquid has passed through the filter, wash the residue upon it with some hot distilled water, letting the washings run into the filtered solution.

Evaporate the liquid in a porcelain dish until a pellicle begins to be formed. Then stir the contents with a glass rod while continuing the evaporation until a granular salt remains. Should this not be perfectly white or colorless, redissolve it in its own weight of boiling distilled water, add a sufficient quantity of ammonia water to render the solution slightly alkaline to test-paper, boil the liquid a few minutes, filter it, and evaporate the filtrate to dryness as before.

Reaction.

$2Fe+2Br_2=2FeBr_2$; then $3FeBr_2+Br_2=FeBr_2.2FeBr_3$; then $FeBr_2.2FeBr_3+8H_4NOH=8H_4NBr+FeO.Fe_2O_3+4H_2O$.

Description.—Colorless, transparent crystals, or a white crystalline granular salt, odorless, of pungent saline taste. Soluble in 1.50 parts of water, and in 30 parts of alcohol at 15° C.; in 0.7 parts of boiling water, and in 15 parts of boiling alcohol.

Another Method.

Bromine ..... 25 parts
Ammonia water.
Water.
Hydrogen sulphide.

Shake 200 parts of water with 5 parts of bromine. Conduct a stream of hydrogen sulphide into the liquid. When the liquid is no longer rendered turbid by the hydrogen sulphide, add another 5 parts of bromine, and again direct hydrogen sulphide into the liquid until the red color disappears. Repeat the addition of bromine, 5 parts at a time, and the treatment with hydrogen sulphide until all of the bromine has been used and converted into hydrobromic acid by the current of hydrogen sulphide.

Filter out the sulphur and heat the filtrate to expel the excess

of hydrogen sulphide from the liquid. When filter paper moistened with a solution of lead acetate is no longer blackened by the liquid, filter again. Supersaturate the filtrate with ammonia (about 60 parts will be required).

Evaporate the solution of ammonium bromide until salt begins to separate. Then add a little more ammonia, after which continue the evaporation to dryness, stirring constantly.

Reaction.

$Br_2+H_2S=2HBr+S$; then

$HBr+H_4NOH=H_4NBr+H_2O$.

AMMONIUM CARBAMATE.

AMMONII CARBAMAS.

$H_4N(H_2NCO_2)=78$.

Dissolve the ammonium carbamate from powdered ordinary ammonium carbonate with strong alcohol and evaporate the alcoholic solution to dryness by the aid of very moderate heat (not exceeding 25°).

Description.—A white powder readily soluble in water and in alcohol.

AMMONIUM CARBONATE—OFFICINAL.

AMMONII CARBONAS OFFICINALIS.

(*Hartshorn Salt.*)

Approximately $H_4NHCO_3.H_4NH_2NCO_2=157$.

This is a somewhat variable mixture of ammonium-hydrogen carbonate and ammonium carbamate.

It is manufactured on a large scale by heating a mixture of ammonium sulphate (or ammonium chloride) and calcium carbonate, when the so-called ammonium carbonate sublimes and is condensed.

Description.—Hard, colorless or white, translucent (nearly transparent in thinner fragments), having, when quite hard and not effloresced, but a faint ammoniacal odor if not confined in a closed vessel. When exposed to the air it becomes covered with a white efflorescence, and finally converted into porous lumps or white powder. Confined in a closed vessel the salt causes the vessel to be filled with vapor of a strongly ammoniacal odor. The taste is sharp and saline.

It is slowly but completely soluble in from 4 to 5 parts of water at 15°. Hot water decomposes it.

Alcohol dissolves out the carbamate, leaving the acid ammonium carbonate as a pulverulent residue.

The reaction of ammonium carbonate is strongly alkaline.

Only *hard, translucent* pieces of ammonium carbonate should be used for pharmaceutical purposes. Pieces covered with a white coating may be scraped, and the hard translucent interior used. When porous and white throughout, the salt should be used only in the production of ammonium salts in cases where the result is not affected by the condition of the ammonium carbonate.

This substance must be kept in tightly closed bottles in a cool place.

AMMONIUM CARBONATE—TRUE.

AMMONII CARBONAS VERUS.

(*Normal or Neutral Ammonium Carbonate.*)

$(H_4N)_2CO_3.H_2O=114.$

Ordinary ammonium carbonate.....	157 parts
Stronger ammonia water.....	63 parts
Distilled water.....	8 parts

Digest them together for two days in a closed wide-mouthed bottle at from 20° to 25°. The whole contents form a solid crystalline mass, which constitutes the di-ammonium carbonate.

Keep the product in a tightly closed bottle in a cool place.

Another Method.

Dissolve eight parts of the ordinary ammonium carbonate in

nine parts of warm ammonia water (containing 10 per cent of H_3N) in a closed bottle. Cool the solution so that crystals may be formed. Collect and drain the crystals quickly and press them gently between cloths or bibulous paper. Bottle at once.

Reaction.

$$H_4NHCO_3.H_4NH_2NCO_2+H_4NOH=2(H_4N)_2CO_3.$$

Description.—Colorless crystals of ammoniacal odor and taste; soluble in their own weight of water at 15°.

AMMONIUM CHLORIDE—PURIFIED.

AMMONII CHLORIDUM PURIFICATUM.

$$H_4NCl=53.4.$$

The impurities liable to be present in crude "sal ammoniac" or crude, commercial ammonium chloride are the chlorides and sulphates of iron and of calcium. The iron compounds may be both ferrous and ferric, but are usually ferrous. Of the impurities mentioned the most common is ferrous chloride. This does not discolor the ammonium chloride until the sal ammoniac is exposed to the air so that the ferrous salt is oxidized to ferric.

The sal ammoniac should be tested for ferrous salt, ferric salt, sulphates and calcium, with test solutions of potassium ferricyanide, potassium ferrocyanide, barium chloride, and ammonium carbonate or oxalate, successively, in the usual way. Should all of the impurities named be present, they may be removed as follows:

Dissolve the impure ammonium chloride in about twice its weight of water, or less. Heat the solution to boiling. Filter. Add solution of barium chloride in small quantities to the hot filtrate and stir well. As soon as a further addition of barium chloride no longer causes turbidity in a filtered test-portion of the liquid, add a solution of ammonium carbonate or oxalate to completely precipitate the calcium and the excess of barium. Filter. Then add to the filtrate about 25 ml of strong chlorine water for each kilogram of ammonium chloride in solution. Boil for twenty or thirty minutes. Then add enough ammonia water to

render the liquid alkaline and to impart an ammoniacal odor to it. Filter again. Evaporate the filtrate to 1.10 sp. w. and then set it aside to crystallize, or evaporate nearly to dryness and dry the granulated salt perfectly in the usual way.

When salts of organic bases are present in the crude sal ammoniac made from gas liquor they should be destroyed by boiling 100 parts of the crude product with 125 parts of water and 15 parts of concentrated nitric acid until no more acid vapors are evolved. The residue may then be treated as before described to remove the chlorides and sulphates of calcium and iron.

Should ferrous chloride be found to be the only impurity present, treat the solution of the impure ammonium chloride with chlorine water and ammonia as described. Should the product be not entirely free from iron after one treatment, repeat the addition of chlorine water, boil again, add ammonia in excess as before and again granulate or crystallize the product.

When ferric chloride is the only impurity the process of purification is simple:

Crude ammonium chloride.....	20 parts
Water	30 parts
Ammonia water (10%).....	1 part

Dissolve the ammonium chloride in the water heated to boiling. Add the ammonia water. Continue the boiling for a few minutes. Filter. Evaporate to granulation. Dry the product thoroughly.

Pure Ammonium Chloride

may be readily made from pure diluted hydrochloric acid and pure ammonia water:

Diluted hydrochloric acid (10% of HCl)....	2 parts
Ammonia water (10% of H_3N).....	1 part

Add the ammonia water gradually to the acid, stirring well. Evaporate to granulation or crystallization, and dry the product.

Reaction. $H_3N+HCl=H_4NCl$.

Description.—Pure ammonium chloride is a colorless crystalline, or a white, granular salt; odorless, taste saline, cooling. Soluble

in 3 parts of water of 15°, and in its own weight of boiling water. Nearly insoluble in alcohol. Reaction neutral. Volatilises without residue when strongly heated.

AMMONIUM IODIDE.

AMMONII IODIDUM.

$H_4NI = 144.50$.

Potassium iodide	6 parts
Ammonium sulphate	4 parts
Alcohol	2 parts
Distilled water	12 parts

Dissolve the salts, each in 6 parts of distilled water; filter the solutions; mix them; evaporate the filtrate to about 15 parts; set the liquid aside in a cool place for about twelve hours; add the alcohol to the solution and set the mixture in an ice-water bath until cooled down to 5°; separate and reject the crystals of potassium sulphate, which are now separated, and add a little ammonia to the clear solution and evaporate it to dryness.

Put the product in a dry bottle, which must be tightly closed, and kept in a dark place.

Reaction. $2KI + (H_4N)_2SO_4 = 2H_4NI + K_2SO_4$.

Notes. The alcohol is added to facilitate the separation of the potassium sulphate, which is insoluble in diluted alcohol, while the ammonium iodide is soluble. The solution may, after standing twelve hours, be cooled to 5° on an ice-water bath before the alcohol is added, the clear solution then separated from the crystals of potassium sulphate by throwing the whole into a cooled glass funnel having a loose plug of moist cotton placed in its throat. The filtrate may then be evaporated to dryness, and the ammonium iodide extracted from the dry salt-mass by means of warm alcohol, which will leave the potassium sulphate undissolved. The alcoholic solution of ammonium iodide may then be allowed to evaporate until the dry salt is obtained. The yield of ammonium iodide from 6 parts of potassium iodide should be about 5.33 parts, which is soluble in 50 parts of warm alcohol.

Description.—A white crystalline powder, without odor, and having a sharp saline taste. Very hygroscopic. Soluble at 15° in two-thirds of its own weight of water and in 9 parts of alcohol; in half its weight of boiling water and in 3.7 parts of boiling alcohol.

The salt is very unstable, soon turning yellow or even brown, and acquiring an odor of iodine. The free iodine by which the product is discolored may be removed by washing the salt with ether and then rapidly drying the white salt; or a concentrated aqueous solution may be made of the salt and enough ammonium sulphide solution added to render the liquid colorless, after which it is filtered and evaporated to dryness.

AMMONIUM NITRATE.

AMMONII NITRAS.

H_4NNO_3=80.

Nitric acid (68%).....	10 volumes
Ammonia water (10% H_3N).....	7 volumes
Distilled water.	

Dilute the nitric acid with 10 volumes of distilled water. Place the ammonia water in a large porcelain dish, and add to it, gradually, the diluted nitric acid, stirring well. When all of the acid has been added, test the reaction of the liquid on litmus paper. If not distinctly (though faintly) alkaline, make it so by the addition of sufficient ammonia water. Filter the solution if not perfectly clear. Evaporate the filtrate until it acquires the density of 1.25 while still hot. Then set it aside to cool that crystals may be formed. Collect and dry the crystals.

Additional crops of crystals may be obtained from the mother liquor by further evaporation.

Keep the product in dry, well closed bottles.

Reaction. $H_4NOH+HNO_3=H_4NNO_3+H_2O$.

Notes. If fused ammonium nitrate is desired instead of crystals, the salt should be fused at a temperature not exceeding 166° C. and should be kept at that temperature until it ceases to give off watery vapor.

During the evaporation of a solution of ammonium nitrate it

happens that the liquid gradually acquires an acid reaction; this should be corrected by the addition of enough ammonia to restore a *slightly* alkaline reaction before the liquid is set aside to crystallize.

Description.—Long, colorless, prismatic crystals, or a fused white mass. Odorless. Taste sharp and bitter. Hygroscopic. Soluble at 15° in half its weight of water or in 20 parts of alcohol; very soluble in boiling water, and in 3 parts of boiling alcohol.

AMMONIUM OXALATE.

AMMONII OXALAS.

$$(H_4N)_2C_2O_4.H_2\dot{O}=142.$$

Oxalic acid	100 Gm
Boiling distilled water	800 ml
Ammonium carbonate, sufficient.	

Dissolve the acid in the water, neutralize with ammonium carbonate, raising the temperature, at the end of the neutralization, to the boiling point; filter while hot, and set aside to cool and crystallize.

Reaction. $2(H_4NHCO_3.H_4NH_2NCO_2)+3H_2C_2O_4 = 3(H_4N)_2C_2O_4+2H_2O+4CO_2.$

Notes. About 83 Gm of ammonium carbonate will be required to saturate 100 Gm of oxalic acid. Instead of 83 Gm of ammonium carbonate, the corresponding quantity (270 ml) of ammonia water (10 per cent) may be used. The solution should be rendered exactly neutral to test-paper, filtered while hot, and set aside to cool slowly. When the solution is dilute and the cooling and crystallization slow, very handsome needle-shaped crystals may be obtained. By evaporating the mother liquor and again crystallizing, additional crops of the salt are recovered.

Description.—This salt crystallizes in rhombic prisms, easily soluble in water.

AMMONIUM PHOSPHATE.

AMMONII PHOSPHAS.

$(H_4N)_2HPO_4=132.$

Prepared by adding strong ammonia solution to diluted phosphoric acid until a slightly alkaline reaction on test-paper is produced. The solution is then evaporated, the alkaline reaction being maintained by adding a little more ammonia from time to time, as required. The crystals formed upon allowing the solution to cool are collected and quickly dried on bibulous paper placed upon a porous tile.

Ammonium phosphate must be kept in well-stoppered bottles.

Description.—Transparent, colorless prisms, inodorous, of a somewhat alkaline saline taste. Readily soluble in water; insoluble in alcohol.

AMMONIUM SULPHATE.

AMMONII SULPHAS.

$(H_4N)_2SO_4=132.$

Ammonia water	2 volumes
Diluted sulphuric acid	5 volumes

Add the acid gradually to the ammonia water, stirring the liquid. Should the solution not be alkaline in its reaction on test-paper, add enough additional ammonia water to render it alkaline. Filter. Evaporate to crystallization. During the evaporation test the liquid from time to time with litmus paper, and add more ammonia if necessary to prevent the solution from acquiring an acid reaction, or to render it alkaline again should it become acid by the loss of ammonia.

Reaction. $2H_4NOH+H_2SO_4=(H_4N)_2SO_4+H_2O.$

Description.—Colorless and odorless crystals, soluble in 1.33 parts of water at 15°.

AMMONIUM VALERATE.

AMMONII VALERIANAS.

$NH_4C_5H_9O_2$=119.

Prepared by saturating valeric acid with dry ammonia gas, usually generated from a mixture of equal parts of ammonium chloride and lime. The heat produced by the chemical union keeps the salt in a liquid condition, and on cooling the valerate crystallizes. The salt cannot be crystallized from an aqueous solution.

A solution of ammonium valerate may be readily prepared by dissolving 102 Gm of valeric acid in 61 Gm of stronger water of ammonia; if diluted with distilled water until it measures 238 milliliters, each ml of this solution will contain 0.50 Gm of the salt.

Neutral ammonium valerate easily parts with ammonia and becomes acid; in solution it is to be carefully neutralized with ammonia water.

Description.—Colorless crystalline plates, soluble in water and alcohol, and having the odor of the acid. They should volatilize completely.

ANTIMONY CHLORIDE.

ANTIMONI CHLORIDUM.

$SbCl_3$=226.5.

Antimonous oxide	3 parts
Hydrochloric acid (32% of HCl)	8 parts

Heat gently together in a porcelain dish, stirring well, until the acid ceases to dissolve any more of the oxide. Then raise the temperature slowly to the boiling point and continue boiling until a drop of the liquid deposits crystals on cooling. Then pour the liquid into a retort provided with a thermometer in the tubulure and distill, heating the retort in a sand bath. The distillate at first consists of hydrochloric acid, which is to be collected separately. As soon as the boiling point rises to 200° and a drop of the distillate solidifies on a cold surface, change the receiver and con-

tinue the distillation. The distillate now passing over is antimony trichloride.

Reaction. $Sb_2O_3+6HCl=2SbCl_3+3H_2O$.

Description.—A white or yellowish-white soft solid or semi-solid. Very deliquescent. Decomposes when brought in contact with water. Soluble in alcohol without decomposition. It can be dissolved in water containing hydrochloric acid, citric acid, or tartaric acid. It is also soluble in glycerin.

Antimony Chloride Solution.

LIQUOR ANTIMONI CHLORIDI.

A solution of $SbCl_3$ in water containing hydrochloric acid.

Purified antimonous sulphide, in extremely fine powder	100 Gm
Hydrochloric acid (35% of HCl)	530 Gm

Put the acid in a flask of one cubic-decimeter's capacity. Heat it to about 50°. Add the antimonous sulphide, *in very fine powder,* gradually to avoid too copious evolution of hydrogen sulphide. Shake well. Apply heat, first gently, then gradually raising the temperature to the boiling point of the liquid. Boil for half an hour. Let the liquid cool somewhat. Filter it through muslin, returning the portion which first passes until the filtrate runs clear. Evaporate the filtrate to 250 ml, and put it in a glass-stoppered bottle.

Reaction. $Sb_2S_3+6HCl=2SbCl_3+3H_2S$.

Notes. Read the notes under the title Antimony Oxide.

Should the antimony sulphide be adulterated and leave an undissolved residue the solution must be correspondingly deficient in strength.

The proportions of antimony sulphide and hydrochloric acid are those prescribed in the British Pharmacopœia of 1885. The quantity of acid is more than twice the quantity required by theory. An excess is necessary to hold the antimony chloride in solution, and a strong acid is necessary to act on the sulphide.

Description.—A yellowish-red liquid of the odor of hydrochloric acid. It gives a white precipitate when mixed with water. Sp. w. about 1.47; sp. vol. 0.680. It can be diluted with 1.25 times its own weight of water without permanent precipitation.

ANTIMONY OXIDE.

ANTIMONI OXIDUM.

Sb_2O_3=287 (or Sb_4O_6=574).

Purified antimonous sulphide, in very fine powder..100 parts
Hydrochloric acid (35% of HCl).....530 parts
Ammonia water (10% of H_3N).....115 parts
Water.

Put the hydrochloric acid in a flask capable of holding 1000 parts of water. Heat to about 50°. Add the finely powdered antimony sulphide, in small portions, shaking well, and waiting after each addition until the evolution of hydrogen sulphide has nearly ceased before adding another portion of the sulphide of antimony. Continue the heat at 50° and add hot water from time to time to maintain the volume of liquid in the flask, or to keep the flask about half filled. When all of the antimony sulphide has been added and the evolution of hydrogen sulphide becomes very slow, heat the contents of the flask to boiling and continue the boiling for half an hour, still adding enough hot water from time to time to keep the volume of the contents of the flask nearly constant. Then let the liquid cool. Filter it through muslin into a porcelain dish. Add just enough water to produce a slight but permanent turbidity. Boil the liquid again for about five minutes. Filter again. Pour the filtrate slowly into 2500 parts of water contained in a glass or stoneware vessel, stirring the mixture diligently during the addition of the antimony solution to the water. Let the precipitate subside. Siphon off the supernatant liquid and add to the precipitate 2500 parts of hot water, stir well, let stand until the precipitate has settled, and then draw off the wash water. Repeat this washing once more, in the same manner, with another 2500 parts of hot water, and siphon off the liquid. Then add the ammonia water to the precipitate, let the mixture stand for an hour, stirring frequently. Collect the precipitate on a filter and

wash it with boiling water until a test-portion of the washings, after acidulation with nitric acid, no longer produces a cloudiness with test-solution of silver nitrate. Then drain the oxide and dry it at a temperature not exceeding 100°.

Reactions. $Sb_2S_3+6HCl=2SbCl_3+3H_2S$;

then $4SbCl_3+5H_2O=2SbOCl.Sb_2O_3+10HCl$; and, finally,

$2SbOCl.Sb_2O_3+2H_4NOH=2Sb_2O_3+2H_4NCl+H_2O$.

Notes. A very large excess of hydrochloric acid is used in order to hold the antimony chloride in soltion. Nevertheless, the action of the acid upon the antimony sulphide is slow. A high temperature is necessary. The operation should be carried out under a hood with a good draught to carry off the vapors of hydrogen sulphide.

Some hydrochloric acid is lost by volatilization during the process of heating it with the antimony sulphide. A hydrochloric acid of less than 35. per cent (1.17 sp. w.) strength does not dissolve the antimony sulphide completely.

The antimonous sulphide should be *pure*, and in very fine ("impalpable") powder. It may be tested before being used, to see that it is soluble in the strong hydrochloric acid without any residue.

The purified antimonous sulphide of the Pharmacopœia, although nearly or quite free from arsenic, may contain some lead. The lead chloride derived from an impure antimony sulphide is washed out from the precipitated "oxychloride of antimony" by means of hot water.

Sodium carbonate may be used instead of ammonia water (using 95 parts of the carbonate in place of 115 parts of ammonia water).

Description.—A heavy, light-grayish, or nearly white, odorless and tasteless powder, insoluble in water and in alcohol. It turns yellow when heated, but becomes white again on cooling.

Another Method.

Heat powdered antimonous sulphide in the air until sulphurous

vapors cease to be given off and a fused, glassy residue is obtained:

$$2Sb_2S_3+9O_2=2Sb_2O_3+6SO_2.$$

The glassy antimonous oxide is translucent and may be reduced to a white powder.

This method of preparation is called "roasting."

ANTIMONY OXYCHLORIDE.

ANTIMONI OXYCHLORIDUM.

(Algaroth's Powder.)

$$2SbOCl.Sb_2O_3=630.8.$$

Prepared by pouring a solution of antimony chloride (see Antimony Chloride Solution and Antimony Oxide) into a large quantity of water. The directions given under the title Antimony Oxide down to and including the washing of the precipitated oxychloride with hot water may be followed (the treatment of the washed precipitate with ammonia being omitted). The product is then dried with the aid of heat.

It is a white, insoluble powder.

ANTIMONYL-POTASSIUM TARTRATE.

ANTIMONI ET POTASSII TARTRAS.

[Tartar Emetic.]

$$2KSbOC_4H_4O_6.H_2O=662.42.$$

Antimonous oxide	4 parts
Potassium bitartrate	5 parts
Water	50 parts

Mix the oxide and the cream of tartar with enough water to form a paste, and set this aside for twenty-four hours. Then add the remainder of the water and boil the mixture in a porcelain capsule for fifteen minutes, stirring frequently, and replacing the water lost by evaporation. Filter while hot, evaporate the filtrate

to 30 parts, and set it aside to crystallize. Wash the crystals with a little cold water, and then dry them between filter paper.

Reaction. $2KHC_4H_4O_6+Sb_2O_3=2K(SbO)C_4H_4O_6+H_2O$.

Notes. A slight excess of antimonous oxide is used, but remains undissolved and is filtered away. To obtain the salt in minute crystals the solution obtained by the above formula may be evaporated down to 27 parts and then shaken in a bottle until cold, when the greater part of the salt will separate. The mother liquor will yield more crystals, but after two or three crystallizations it becomes quite colored, as is the case in making several other tartrates.

Description.—Small, transparent crystals, or white powder, odorless, having a sweet, afterwards disagreeable, metallic taste. Soluble in 17 parts of water at 15°, and in 3 parts of boiling water. Insoluble in alcohol.

The colorless crystals become white or opaque on exposure to the air.

ANTIMONIC SULPHIDE.

ANTIMONI PENTASULPHIDUM.

(*Stibium Sulphuratum Aurantiacum.*—"*Golden Sulphuret of Antimony.*")

Chiefly $Sb_2S_5=400$.

Purified black antimony sulphide.....	18 parts
Sublimed sulphur	5 parts
Sodium hydroxide	13 parts
Water	50 parts

Sulphuric acid, distilled water, each sufficient.

Triturate the antimonous sulphide and the sulphur together until mixed; add the mixture to the sodium hydroxide previously dissolved in the water; boil the mixture with constant stirring about fifteen minutes, or until no more of the powder dissolves. Filter, evaporate the filtrate, and let it cool, that crystals may form.

Rinse the crystals hastily with a little weak soda solution, and dry them by pressing them between bibulous paper. [These crystals are the so-called "Schlippe's Salt"—$Na_3SbS_4+9H_2O$.]

Dissolve 100 parts of these crystals in 400 parts of distilled water. Filter, if necessary. Dilute the filtrate with six hundred parts of distilled water, and pour the dilute solution slowly and with constant stirring into a mixture of thirty-six parts (by weight) of sulphuric acid and six hundred parts of distilled water.

Wash the precipitate as rapidly as possible, by affusion and decantation, with distilled water, let drain, express the remainder of the water, and dry the precipitate with the aid of gentle heat. Keep it in a tightly corked bottle, protected from light.

Reaction.

$$4Sb_2S_3+4S_2+18NaOH=5Na_3SbS_4+3NaSbO_3+9H_2O;$$

then, $2Na_3SbS_4+3H_2SO_4+10H_2O$

$$=Sb_2S_5+3Na_2SO_4.10H_2O+3H_2S.$$

Notes. The direction to wash the crystals of Schlippe's salt is given for the purpose of removing sulpharsenate, which is more readily soluble, and a little sodium hydroxide is added to the wash-water in order to prevent oxidation.

The sulphuric acid must be in excess; otherwise the product will be dark colored.

The washing must be done with distilled water, and should not be long continued. Affusion and decantation is therefore to be preferred.

Several formulas prescribe an excessive proportion of sulphur —equal weights of sulphur and antimony sulphide.

Description.—An orange-red, odorless, insoluble powder.

This preparation is identical with the "sulphurated antimony" of the British Pharmacopœia and with the old "golden sulphuret of antimony." It is redder than the sulphurated antimony of the Pharmacopœia of the United States and the Kermes Mineral of the old pharmacopœias, both of which contain Sb_2S_3 but not Sb_2S_5.

ANTIMONOUS SULPHIDE.

ANTIMONI SULPHIDUM NIGRUM.

Sb_2S_3=336.

Native antimony trisulphide (antimonite), purified by fusion, and as free from arsenic as it can be obtained, constitutes the "antimony sulphide" of the Pharmacopœia.

Description.—Dark steel-gray, striated crystalline masses, of metallic lustre, forming a black or grayish-black lustreless powder when reduced to a very fine state of division; odorless; tasteless; insoluble in water and in alcohol. Soluble in *strong* hydrochloric acid with evolution of hydrogen sulphide.

Purified Antimony Sulphide.

Antimony sulphide	2 parts
Water of ammonia	1 part

Reduce the antimony sulphide to a very fine powder. Separate the coarser particles by elutriation, and, when the finely divided sulphide has been deposited, pour off the water, add the water of ammonia, and macerate for five days, agitating the mixture frequently. Then let the powder settle, pour off the water of ammonia, and wash the residue by repeated affusion and decantation of water. Finally, dry the product by the aid of heat.

Note. By this treatment all but traces of arsenic is removed as soluble ammonium sulph-arsenite.

Description.—An impalpable, heavy, grayish-black, lustreless, odorless, tasteless, insoluble powder.

Sulphurated Antimony.

Chiefly antimonous sulphide with a very small amount of antimonous oxide.

Purified antimony sulphide	1 part
Solution of sodium hydroxide	12 parts
Diluted sulphuric acid, sufficient.	

Mix the antimony sulphide with the solution of sodium hydroxide and 30 parts of distilled water, and boil the mixture gently for two hours, constantly stirring, and occasionally adding distilled water so as to preserve the same volume. Strain the liquid immediately through a double muslin strainer, and drop into it, while yet hot, diluted sulphuric acid so long as it produces a precipitate.

Wash the precipitate with hot distilled water until the washings are at most but very slightly clouded by test solution of barium chloride; then dry the precipitate at 25° and rub it to a fine powder.

Reaction.

First, $Sb_2S_3+6NaOH=Na_3SbO_3+Na_3SbS_3+3H_2O$; then $2Na_3SbO_3+3H_2SO_4=3Na_2SO_4+Sb_2O_3+3H_2O$, and $2Na_3SbS_3+3H_2SO_4=3Na_2SO_4+Sb_2S_3+3H_2S$.

Notes. The antimony sulphide must be in the form of an "impalpable powder." This product is *not* the same as the "sulphurated antimony" of the British Pharmacopœia; the B.P. preparation consists mainly of Sb_2S_5 (see Antimonic Sulphide); neither is it the "Kermes Mineral" of the old pharmacopœias. The Pharmacopœia of the United States, nevertheless, gives this preparation the title of the British official precipitated antimony sulphide as well as the synonym "Kermes Mineral."

Description.—A reddish-brown or brown-red odorless, tasteless, insoluble powder. It is redder than Kermes Mineral, but not so red as the antimonic sulphide or sulphurated antimony of the British Pharmacopœia.

Oxysulphurated Antimony.

(KERMES MINERAL. KERMES ANTIMONI.)

Purified antimony sulphide	1 part
Sodium carbonate	25 parts

Put the sodium carbonate, together with 250 parts of water, into an iron pot, and heat to boiling. Add the sulphide and continue boiling for two hours, stirring constantly, and replacing the

water lost by evaporation. Filter, as rapidly as possible, and while still hot, into a warm porcelain dish or stone jar kept in hot water, so that the filtrate will cool very slowly.

When cold, decant the clear liquid from the precipitate, collect the latter at once on a filter, and wash it with cold distilled water until the washings no longer give an alkaline reaction on test-paper, or until the washings begin to be colored.

Then press the precipitate between blotting paper, and afterwards dry it in a dark place at a temperature not exceeding 30° C. (86° F.). When dry, powder it, and keep it in tightly closed bottles, protected from light.

Reaction.

$$5Na_2CO_3+6Sb_2S_3=NaSbO_2+9NaSbS_2+Sb_2O_3+5CO_2;$$

and $$Na_2CO_3+Sb_2O_3=2NaSbO_2+CO_2.$$

Notes. The product consists mainly of Sb_2S_3 with some Sb_2O_3. This is accounted for by the fact that the hot solution of sodium sulphantimonite ($NaSbS_2$) is capable of dissolving a considerable quantity of antimonous sulphide, which deposits on cooling. At the same time it requires a very large excess of sodium carbonate to prevent the separation of antimonous oxide and sodium of antimonite; hence the quantity of sodium carbonate prescribed is more than twenty times the amount required by theory. Nevertheless the precipitation of antimonous oxide is only partially prevented. If the proportion of sodium carbonate used be diminished the product contains more oxide. The large quantity of water used is also necessary to prevent the separation of an undue proportion of oxide.

The boiling should not be continued longer than the prescribed period, and the solution must not be allowed to cool rapidly. It is best to let the temperature fall gradually from the boiling point down to about 40° during a period of several hours.

If the solution be cooled below 40° the precipitate afterwards formed contains more oxide. Therefore the kermes slowly deposited from the dilute solution during the slow cooling from 100° to 40° is all that can be obtained. It is a very small quantity, and the process is accordingly attended with great waste of material; *but a precipitated antimonous sulphide obtained in any other way is not kermes.*

If sodium hydroxide be used instead of the carbonate, the product is nearly the same in composition, but has an altogether different appearance.

Kermes mineral does not deserve a place in any pharmacopœia, and very few pharmacopœias now contain it. It is a relic of the times when peculiar physical properties due to extraordinary methods of preparation were, often without reason, held to be indicative of extraordinary medicinal virtues.

Description.—Prepared carefully in strict obedience to the directions given, kermes mineral is a beautiful, velvety, dark purplish-brown powder. Minute crystals of antimonous oxide may be seen under the microscope as shining particles in the product.

The Pharmacopœia of the United States (1890) gives the synonym "Kermes Mineral" to the antimonous sulphide precipitated from a solution of sodium sulphantimonite by the addition of sulphuric acid; but that is *not* the kermes mineral of the old pharmacopœias.

The Norwegian Pharmacopœia directs the preparation of kermes mineral by mixing 1 part of antimonous oxide with nine parts of precipitated sulphide.

ARSENOUS ACID SOLUTION.

LIQUOR ACIDI ARSENOSI.

Arsenous oxide	10 Gm
Hydrochloric acid	50 ml
Distilled water, sufficient.	

Boil the arsenous oxide with the acid diluted with 250 ml of the water until dissolved. Filter the solution, pass enough distilled water through the filter to make the product measure 1 liter, and mix the whole well.

Notes. The presence of hydrochloric acid materially aids the solution of the arsenous oxide. When dissolved in the water the oxide is probably converted into arsenous acid:

$$As_2O_3+3H_2O=2H_3AsO_3.$$

This preparation was formerly erroneously called "solution of

chloride of arsenic;" it probably contains very little arsenous chloride.

Description.—A colorless solution with an acid reaction. It gives a bright yellow precipitate with hydrogen sulphide.

ARSENOUS IODIDE.

ARSENI IODIDUM.

AsI_3=454.5.

Arsenic	3 parts
Iodine	16 parts

Triturate until finely powdered and thoroughly mixed. Put into a flask and heat gently on a water-bath, inclining the flask in different directions, until fusion takes place. Pour the fused mass upon a porcelain slab and let it cool, then break it into pieces and keep it in well stoppered bottles in a cool place.

Description.—The product consists of glossy orange-red crystalline masses or scales having an odor of iodine. It decomposes on exposure to air and light.

Solution of Arsenic and Mercuric Iodide.

DONOVAN'S SOLUTION.

Arsenic iodide	1 part
Mercuric iodide	1 part
Distilled water, sufficient.	

Triturate the iodides with 15 parts of distilled water, until dissolved. Filter the liquid and pass enough distilled water through the filter to make the product weigh 100 parts.

Keep the product in a dark place.

Notes. No chemical reaction takes place in dissolving the two iodides together; but the mercuric iodide is soluble in the solution of arsenic iodide, although almost insoluble in water.

The arsenic iodide must not contain free iodine. The preparation does not remain unchanged a long time. It must be odorless and only pale yellowish—not having any odor of iodine nor a

reddish color. As soon as it acquires the odor of iodine or a reddish color it should be thrown away.

Description.—A clear pale yellowish liquid without odor, but having a nauseous metallic taste.

ARSENOUS OXIDE.

ARSENI OXIDUM.

$As_4O_6=396.$

(*Acidum Arsenosum of the Pharmacopoeias.*)

A heavy solid, occurring either in transparent colorless glassy masses, or white porcelain-like, or in white powder; very poisonous.

For medicinal uses it must be in extremely fine powder. It may be powdered by trituration in a porcelain mortar; but it should be kept moist with alcohol while triturated to prevent the rising of the poisonous dust.

Forms a permanent solution in 80 parts of water. Dissolves slowly unless the solution is aided by heat.

BARIUM ACETATE.

BARII ACETAS.

$Ba(C_2H_3O_2)_2.H_2O=273.$

Barium carbonate	10 parts
Acetic acid (36%)	17 parts
Water	10 parts

Dissolve, filter, and evaporate to crystallization. Dry the crystals in a desiccator over sulphuric acid, or with the aid of heat.

Keep the product in a tightly closed bottle.

Description.—White, deliquescent, odorless; readily soluble.

BARIUM BROMIDE.

BARII BROMIDUM.

$BaBr_2.2H_2O=332.$

This salt is prepared by saturating hydrobromic acid with barium carbonate, filtering the solution, and evaporating to crystallization.

It may also be made from barium hydroxide with ammonium bromide in the same manner as calcium bromide is prepared from lime and ammonium bromide.

Another method is analogous to that employed for the preparation of ammonium bromide from bromide of iron, the materials being ferroso-ferric bromide and barium hydroxide.

Description.—Colorless crystals, or a white granular salt; odorless; taste acrid, nauseous. Readily soluble in water and in alcohol.

BARIUM CARBONATE.

BARII CARBONAS.

$BaCO_3=197.$

Barium nitrate	100 parts
Ammonium carbonate	37 parts
Ammonia water (10% of H_3N)	80 parts
Water.	

Dissolve the barium nitrate in 600 parts of water, and filter if necessary. Dissolve the ammonium carbonate in 80 parts of water, and filter if required. Warm the barium nitrate solution to 50° and add it gradually to the ammonium carbonate solution, stirring well. Let the mixture become cold. Then add ammonia water until the mixture acquires a decided ammoniacal odor. Stir well. Let settle. Decant the liquid. Wash the precipitate until the washings are free from ammonium salts (as indicated by their being no longer affected by sodium cobaltic nitrate test-solution). Dry the precipitate with the aid of heat.

Reaction.

$$Ba(NO_3)_2+(H_4N)_2CO_3=BaCO_3+2H_4NNO_3.$$

Notes. Ammonium carbonate is prescribed instead of potassium or sodium carbonate, because it is scarcely possible to remove all potassium or sodium salt from the precipitated barium carbonate, while ammonium salt is easily removed. Ammonia water is added not only to convert the ordinary carbonate into the normal salt, but also because the barium carbonate is somewhat soluble in ammonium nitrate solution, but not in the ammoniacal liquid obtained by the addition of an excess of ammonia.

Description.—A heavy, white, insoluble, odorless and tasteless powder. Completely soluble in nitric acid and in hydrochloric acid.

Other Methods.

From heavy spar (barium sulphate) the carbonate of barium can be obtained by heating to redness an intimate mixture made of 10 parts of powdered heavy spar, 2 parts of powdered charcoal, and 5 parts of potassa, and then washing the fused mass with water, when the undissolved residue is barium carbonate.

$$BaSO_4+2KOH+2C=BaCO_3+K_2S+H_2O+CO_2.$$

The impure native barium carbonate called "witherite" may be converted into chloride as described under the title Barium Chloride, and the pure carbonate then made from the chloride by precipitation with ammonium carbonate.

BARIUM CHLORIDE.

BARII CHLORIDUM.

$$BaCl_2.2H_2O=243.56.$$

Powdered witherite..... 10 parts
Hydrochloric acid (31.9% of HCl)..... 11 parts
Barium sulphide.
Water.

Mix the witherite thoroughly with its own weight of water in

a porcelain dish. Then add, gradually, the hydrochloric acid previously mixed with 10 parts of water, stirring well, waiting after each addition of the dilute acid until the effervescence has subsided before adding more. When the interaction becomes tardy, heat the dish over the water-bath to facilitate the saturation of the acid. When all of the acid has been added, evaporate the unfiltered liquid to dryness, and heat the dry residue at about 100° for half an hour. Then add 50 parts of boiling water; stir well, continuing the heating and stirring about five minutes. Filter. Add to the filtrate a solution of barium sulphide until no further precipitation is produced by it. Filter again. Acidify the filtrate with hydrochloric acid so that it just shows a distinct acid reaction on litmus paper. Then evaporate the solution to the density of about 1.3 and set it aside in a cool place to crystallize.

Recrystallize the salt two or three times, as may be necessary.

Reaction. $BaCO_3+2HCl=BaCl_2+H_2O+CC_2$.

Notes. The mineral "witherite" is an impure native barium carbonate, containing varying amounts of calcium, strontium and iron compounds together with other mineral substances. A very large excess of witherite must necessarily be used, because much of it may consist of substances other than barium carbonate, and it is further requisite, that, after the saturation of the acid, a considerable amount of barium carbonate shall still remain in order that, during the evaporation of the solution to dryness, the iron and calcium may be precipitated. The remaining iron and any other heavy metals present are removed by precipitation with barium sulphide, which must, however, be employed with great caution to avoid using an excess. The final solution still contains chlorides of the alkali metals and of calcium and strontium, which must be removed by re-crystallization of the barium chloride which is less freely soluble than the others, so that the latter remain in the mother liquors. The concentrated solution of barium chloride can also be mixed with alcohol, which precipitates the salt while the calcium and strontium chlorides remain in solution.

A pure barium chloride may, of course, be at once obtained by saturating pure hydrochloric acid with pure barium carbonate.

Description.—Colorless crystals, soluble in 3 parts of water at 10°, in 2 parts at 72°, and in 1.6 parts of boiling water. The solution is neutral.

BARIUM CHROMATE.

BARII CHROMAS.

$BaCrO_4$=253.

Barium acetate..... 50 parts
Potassium dichromate..... 27 parts
Water.

Dissolve the dichromate in 75 parts of hot water and filter. Dissolve the barium acetate also in 75 parts of water and filter. Add the latter solution to the former, stirring well. Wash the precipitate by affusion and decantation of cold water. Collect, drain and dry it.

Reaction.

$$2Ba(C_2H_3O_2)_2.H_2O+K_2Cr_2O_7 = 2BaCrO_4+2KC_2H_3O_2+2HC_2H_3O_2.$$

Description.—An insoluble lemon-yellow powder, used as a pigment.

BARIUM DIOXIDE.

BARII DIOXIDUM.

BaO_2=169.

When a current of air is conducted over barium oxide heated at 450° C. the oxide is converted into dioxide. The barium dioxide is employed in the preparation of hydrogen dioxide.

Description.—A heavy, grayish-white, or pale yellowish-white, coarse powder. Odorless and tasteless. Nearly insoluble in cold water.

BARIUM NITRATE.

BARII NITRAS.

$Ba(NO_3)_2$=261.

Powdered witherite..... 16 parts
Nitric acid (68% of HNO_3)..... 15 parts
Water.

Mix the witherite thoroughly in a porcelain dish with its own weight of water. Dilute the nitric acid with its own weight of water. Add the diluted acid, in small portions, to the witherite, stirring well and waiting after each addition until the effervescence has subsided before adding the next portion of acid. When about one-half of the acid has been added, place the dish over a water-bath, apply heat, add about 40 parts of hot water, stir well and then proceed with the addition of the remainder of the nitric acid, in small portions, as before, stirring well. Evaporate the contents of the dish to dryness. Add to the dry residue 60 parts of boiling water, stir well, and continue heating the mixture at 90° or over for half an hour. Filter. Acidify with nitric acid. Evaporate the filtrate to crystallization. Recrystallize two or three times, as may be necessary.

Reaction. $BaCO_3+2HNO_3=Ba(NO_3)_2+H_2O+CO_2$.

Notes. The impure barium carbonate is used in large excess (see notes under Barium Chloride). Iron is precipitated by the excess of barium carbonate. The nitrates of strontium and calcium remain in the mother-liquors when the product is re-crystallized.

The crystallization proceeds most satisfactorily when the acidified solution saturated at about 80° to 90° is slowly cooled.

Pure barium carbonate and nitric acid readily produce a pure barium nitrate.

Description.—Colorless or white crystals. Soluble in 7 parts of water at 10°; in 9.2 parts at 20°; in 5 parts at 60°; and in a little less than 3 parts of boiling water.

BARIUM HYDROXIDE.

BARII HYDROXIDUM.

$Ba(OH)_2$=171.

Barium chloride	1,000 parts
Sodium hydroxide	310 parts
Water.	

Dissolve the barium chloride in 2,000 parts of hot water and the sodium hydroxide in 840 parts of water. Mix the solutions. Heat to boiling. Filter while hot. Set aside to crystallize. Recrystallize the hydroxide twice from hot water. Drain the crystals on a platinum cone by means of a filter pump. Preserve the product in tightly stoppered bottles.

BARIUM OXIDE.

BARII OXIDUM.

BaO=153.

Prepared by heating barium nitrate to white heat in a Hessian crucible covered with a fire clay cover. The Roessler furnace may be used for this purpose when the quantity is small.

Properties. Grayish-white, porous pieces, forming barium hydroxide with water.

BISMUTH; PURIFIED.

BISMUTHUM PURIFICATUM.

Bi=208.

Commercial bismuth	1 part
Potassium nitrate	11 parts

Triturate the bismuth with ten parts of the potassium nitrate until the two substances are well mixed. Heat the mixture in a crucible to complete fusion, stir uninterruptedly with a clay rod (or pipe-stem) and keep the mass in a state of fusion for at least half an hour. Then pour the fused mass into a vessel of

hot water. Dry the undissolved residue, consisting of bismuth and some bismuth oxide, mix it well with 1 part of potassium nitrate, and fuse the mixture as before, and separate the slag from the insoluble bismuth oxide and the ingot of metal.

Notes. Commercial bismuth contains arsenic, sulphur and selenium. They are converted into arsenate, sulphate and selenate of potassium by fusion with the potassium nitrate. These salts together with the excess of potassium nitrate are water-soluble, and thus separated by the water. The bismuth is fused together in one lump. Any bismuth oxide formed remains in pulverulent form when the slag of salts has been washed off, and can be recovered.

Another Method.

Bismuth	2 parts
Sodium nitrate	1 part
Solution of sodium hydroxide (5% of NaOH)	9 parts
Water.	

Mix the bismuth and sodium nitrate and heat the mixture in an iron dish at low red heat. When the mass begins to swell stir uninterruptedly with an iron spatula for about an hour or until the metal is finely divided and thus scarcely distinguishable in the mixture. Then remove the dish from the source of heat, and add to the half-cooled contents the solution of sodium hydroxide. Heat the mixture to boiling, stirring well. Transfer the whole to a paper filter, let the liquid run off, wash the finely divided bismuth remaining on the filter, and dry it.

Some bismuth oxide is mixed with the metal thus purified, and it is possible that some bismuth arsenate may also remain with the product.

BISMUTH BENZOATE.

BISMUTHI BENZOAS.

Freshly prepared and still moist bismuth hydroxide is digested at about 80° C. with an excess of benzoic acid mixed with ten times its weight of water in a porcelain dish over a water-bath for about half an hour.

[The quantity of bismuth hydroxide required for 100 Gm of benzoic acid is about that corresponding to 175 Gm of bismuthous oxide.]

The insoluble product (which is said to yield about 64 to 65 per cent of bismuthous oxide on ignition) is washed as rapidly as possible with cold water, and dried at a temperature not exceeding 80° C.

Another Method.

Crystallized normal bismuth nitrate	20 parts
Glycerin	30 parts
Sodium benzoate	20 parts
Distilled water, sufficient.	

Crush and then powder the bismuth nitrate in a deep porcelain dish. Pour upon it the glycerin and stir well together. Place the dish over a water-bath and heat until the bismuth nitrate is dissolved. Add 65 parts of distilled water.

Dissolve the sodium benzoate in one liter of water at a temperature of about 40° to 50° C.

Add the bismuth solution gradually to the solution of sodium benzoate, stirring well. Let the precipitate subside, decant the mother-liquor, and wash the precipitate with warm water (40° —50° C.) by decantation until the washings give no further reaction for nitrate. Dry the product, first at the ordinary temperature and finally at not over 80° C. Reduce it to fine powder.

Description.—A white, amorphous, odorless and tasteless powder, insoluble in water. Decomposed by hydrochloric acid or by nitric acid with the separation of benzoic acid.

BISMUTH CITRATE.

BISMUTHI CITRAS.

$BiC_6H_5O_7$=397.

Bismuth subnitrate	10 parts
Citric acid	7 parts

Boil them with 40 parts of distilled water until a drop of the mixture yields a clear solution with ammonia water. Add 500

parts of distilled water, stir well, and then set the mixture aside to settle. Decant the acid supernatant liquid, and wash the precipitate, first by decantation and afterwards on a filter, until the washings are tasteless. Dry the product by moderate heat.

Reaction.

$$OBiNO_3.H_2O+H_3C_6H_5O_7.H_2O=BiC_6H_5O_7+HNO_3+3H_2O.$$

Notes. Citrate of bismuth is insoluble in water, and nearly so in the dilute nitric acid formed by the reaction taking place in this process. Subnitrate of bismuth is insoluble in ammonia water, but the citrate is soluble in it, and hence the reaction is known to have been completed when the turbid liquid is cleared by ammonia water.

Another Method.

Subnitrate of bismuth	22 Gm
Citric acid	16 Gm
Sodium bicarbonate	32 Gm
Nitric acid, 44 ml, or sufficient.	
Distilled water, sufficient.	

Heat the subnitrate of bismuth with the nitric acid until dissolved. Add a little water, with constant stirring, until the cloudiness produced by the water no longer rapidly disappears. Dissolve the sodium bicarbonate in 480 ml of distilled water, add the citric acid, boil until effervescence ceases, and then add this solution to the clear (or only faintly opalescent) solution of nitrate of bismuth until no further precipitate is produced. Heat the mixture to boiling, stirring occasionally. Set aside to cool. When cold, filter, and wash the precipitate until all free nitric acid has been removed. Dry the product by water-bath heat.

Reaction. Bismuth nitrate is formed by the solution of the subnitrate in nitric acid; the citric acid and sodium bicarbonate yield sodium citrate. The reaction between these salts is:

$$Na_3C_6H_5O_7+Bi(NO_3)_3=BiC_6H_5O_7+3NaNO_3.$$

But nitric acid is used in excess, and sodium bicarbonate is also used in excess, and the excess of acid on the one hand is nearly neutralized by the excess of alkali on the other.

Description.—A white, odorless and tasteless powder, readily soluble in ammonia water, but insoluble in water and in alcohol. Soluble in solutions of alkali citrates.

Bismuth and Ammonium Citrate.

BISMUTHI ET AMMONII CITRAS.

Citrate of bismuth..... 1 part
Water of ammonia, sufficient.

Mix the citrate of bismuth with two parts of distilled water to a smooth paste, and gradually add water of ammonia until the salt is dissolved, and the liquid has a neutral or only faintly alkaline reaction. Then filter the solution, evaporate it to a syrupy consistence, spread it on plates of glass, and dry it in scales. Keep the product in small, well stoppered bottles, protected from light.

Description.—Small scales, soluble in water. Odorless. Taste slightly acidulous and slightly metallic. On being kept for some time it becomes only partially soluble in water; but the addition of ammonia effects complete solution.

BISMUTH HYDROXIDE.

BISMUTHI HYDROXIDUM.

$Bi(OH)_3$=260.

Crystallized normal bismuth nitrate..... 10 Gm
Nitric acid..... 13 Gm
Distilled water, ammonia water, each sufficient.

Dissolve the bismuth nitrate in the nitric acid and 100 Gm of water previously mixed. Precipitate with ammonia in excess, by pouring the bismuth solution gradually and with constant stirring into 35 Gm of ammonia water. Decant the liquid from the precipitate. Add to the precipitate 100 Gm of distilled water mixed with 10 Gm of ammonia water and let the mixture stand for a few hours, to decompose any subnitrate which may be contained in the precipitate. Wash the product by affusion and decantation of cold distilled water until the washings give no

further reaction for nitrate and leave no residue on evaporation. Dry the product without heat.

Reaction.

$$OBiNO_3.H_2O+H_4NOH=H_4NNO_3+Bi(OH)_3.$$

Description.—A white, fine, odorless and tasteless powder.

BISMUTH NITRATE.

BISMUTHI NITRAS.

(*Trisnitrate of Bismuth.*)

$$Bi(NO_3)_3.5H_2O=484.$$

Proceed as in the formula for making subnitrate of bismuth up to the point where the precipitated subcarbonate has been redissolved in nitric acid. Evaporate this solution to crystallization. Dry the crystals by gently pressing them between white filter paper, and at once put them in a glass stoppered bottle.

Description.—The salt is in large, transparent crystals, colorless if pure. Decomposed by water; soluble in dilute nitric acid, and in glycerin, also in glacial acetic acid.

Glycerite of Bismuth Nitrate.

Normal bismuth nitrate	1 part
Glycerin	8 parts

Powder the crystals by trituration in a mortar. Then add the glycerin, all at once, and stir until the salt has dissolved.

Notes. If the crystals are triturated *with* the glycerin, especially with a *small quantity* of glycerin, there is danger that nitroglycerin may be formed and explosion result.

The solution is used for preparing certain other bismuth preparations by double decomposition, as the oleate, salicylate, and valerate.

BISMUTH OLEATE.

BISMUTHI OLEAS.

$Bi(C_{18}H_{33}O_2)_3 = 105.$

Normal bismuth nitrate	20 Gm
White Castile soap, in fine powder	32 Gm

Put the crystals of normal nitrate of bismuth into a mortar, powder the salt, and then add, all at once, 100 ml of glycerin; stir occasionally, avoiding friction or pressure, until the nitrate is dissolved, which will be accomplished in a few hours. Dissolve the soap in 1,500 ml of water, and then add slowly the solution of nitrate of bismuth. Warm the mixture, reject the watery mother-liquor, and wash the oleate twice with warm distilled water.

Reaction.

$$Bi(NO_3)_3 + 3NaC_{18}H_{33}O_2 = Bi(C_{18}H_{33}O_2)_3 + 3NaNO_3.$$

Notes. The normal nitrate of bismuth is to be powdered before adding the glycerin, and the glycerin is added all at once to prevent possible explosion of nitro-glycerin, which might be formed. This oleate must be fused by very gentle heat, over a water-bath, to get rid of the last portions of water. The yield is about 36 Gm, and the product is a white or yellowish white ointment containing about 20 per cent of bismuth.

BISMUTH OXIDE.

BISMUTHI OXIDUM.

$Bi_2O_3 = 466.$

Bismuth subnitrate	1 part
Solution of sodium hydroxide (5% of NaOH)	4 parts

Mix in a porcelain dish and boil the mixture for ten minutes.

Set it aside to settle. Wash the precipitate with hot water several times. Dry it.

Reactior

$$2BiONO_3.H_2O+2NaOH=Bi_2O_3+2NaNO_3+2H_2O.$$

Description.—A fine, soft, white, odorless and tasteless powder.

BISMUTH SALICYLATE.

BISMUTHI SALICYLAS.

Prepared by double decomposition from normal bismuth nitrate (59 parts) and sodium salicylate (51 parts). The nitrate of bismuth is dissolved in glycerin, and this solution gradually added to a strong solution of the sodium salicylate in water.

Another Method.

Salicylic acid is mixed with ten times its weight of water in a porcelain dish; bismuth hydroxide, recently prepared and not dried, is added, and the mixture heated at about 80° C. The salicylic acid must be used in excess, and the liquid must at the end remain acid in reaction. Continue heating the mixture for half an hour, stirring constantly. Let it cool. Wash the precipitate on a strainer or in a paper filter, according to the quantity operated upon, with cold distilled water, as rapidly as practicable, and dry the product at not over 80° C.

Description.—Salicylate of bismuth is of indefinite composition. It consists of a white, odorless powder, almost entirely insoluble in water, but soluble in nitric acid or hydrochloric acid with separation of salicylic acid.

The preparation yields all of its salicylic acid to boiling alcohol, or to cold ether.

It should contain an amount of bismuth corresponding to about 61 per cent of bismuthous oxide.

BISMUTHYL CARBONATE.

BISMUTHI SUBCARBONAS.

[*Subcarbonate of Bismuth.*]

$(BiO)_2CO_3.H_2O=526.$

Bismuth, in small pieces	50 Gm
Nitric acid	150 ml
Ammonia water	125 ml
Sodium carbonate	250 Gm

Mix 800 ml of the nitric acid with 100 ml of distilled water in a capacious glass vessel, add the bismuth, and set aside for twenty-four hours to dissolve. Dilute the solution with 250 ml of distilled water (until permanent turbidity results), stir well again, set the liquid aside for twenty-four hours, and then filter. Dilute the filtrate by gradually adding to it 1,600 ml of distilled water, and mixing well. Now add, slowly, the ammonia water through a small siphon, stirring constantly and actively. Transfer the whole to a wetted muslin strainer, and let drain. When the liquid has drained off pour upon the precipitate 800 ml of distilled water, mixing well, and then let drain again. Now transfer the precipitate to a suitable vessel, add the remainder of the nitric acid, and, when all has dissolved, also add gradually 100 ml of distilled water, and set the solution aside for twenty-four hours. Then filter.

Dissolve the sodium carbonate in 300 ml of distilled water, with the aid of heat, filter the solution, and set it aside until cold. Now pour the cold solution of bismuth nitrate very slowly into the solution of sodium carbonate, with constant and brisk stirring. Transfer the whole to a wetted muslin strainer, and, after draining thoroughly, wash the precipitate with distilled water, as expeditiously as practicable, until the washings become tasteless. Lastly, press out the moisture as far as practicable, dry the precipitate on bibulous paper, or on muslin, at a gentle heat, and then powder it by rubbing it through a fine sieve by means of a soft brush.

Reaction. First a solution of bismuthous nitrate is made:

$$Bi+4HNO_3=Bi(NO_3)_3+2H_2O+NO.$$

This is poured into a large quantity of water containing ammonia, whereby bismuthyl nitrate is thrown down, the ammonia being added to neutralize the free nitric acid so as to prevent it from retaining any bismuth nitrate in the liquid. Then the precipitate is redissolved in nitric acid to form again the $Bi(NO_3)_3$.

Finally, $2Bi(NO_3)_3+3Na_2CO_3.H_2O$
$=(BiO)_2CO_3.H_2O+6NaNO_3+2CO_2$.

Notes. As the bismuth usually contains arsenic the acid solution first prepared contains bismuth arsenate; when the liquid is diluted with water until permanently somewhat turbid, the arsenical salt precipitates before the bismuthyl nitrate does, and hence, after twenty-four hours' rest, most of the arsenate is found deposited as a white precipitate, and is then filtered away. The rest of the arsenic remains in the mother-liquor after the precipitation of the bismuth nitrate with ammonia, as ammonium arsenate. See also the notes under the title Bismuthyl Nitrate.

Description.—A soft, white or pale yellowish, odorless and tasteless powder, which becomes converted into oxide by heat. It is quite insoluble in water, but should give a clear solution with nitric acid.

Another Method.

Purified bismuth, in small pieces	100 Gm
Nitric acid	200 ml
Ammonium carbonate	300 Gm
Distilled water, sufficient.	

Mix the nitric acid with 150 ml of distilled water, and add the bismuth in successive portions. When effervescence has ceased, apply for ten minutes a temperature approaching that of ebullition. Then decant the solution from any insoluble matter that may be present. Evaporate the solution until it is reduced to 100 ml, and add this in small quantities at a time to a cold filtered solution of the ammonium carbonate in 2 liters of distilled water, stirring constantly during the admixture. Collect the precipitate on a filter and wash it with distilled water until the washings pass tasteless. Remove now as much of the adhering water as can be separated from the precipitate by slight pressure with the

hands, and finally dry the product at a temperature not exceeding 60.°5 C.

Description.—A soft, white, odorless and tasteless powder, insoluble in water but completely soluble in dilute nitric acid, with effervescence.

BISMUTH SUBGALLATE.

BISMUTHI SUBGALLAS.

Crystallized normal bismuth nitrate	100 Gm
Glacial acetic acid	200 Gm
Gallic acid	33 Gm

Dissolve the bismuth nitrate in the acetic acid and dilute the solution with 500 Gm of distilled water. Filter. Add gradually to this liquid a solution of the gallic acid in 1,500 Gm of cold distilled water. Wash the precipitate with tepid distilled water until the washings are free from nitric acid. Dry the product at from 70° to 80° C. on blotting paper spread upon porous tiles.

Description.—A sulphur yellow, odorless and tasteless powder; insoluble in water or in alcohol. Probably practically identical with the preparation called "dermatol."

BISMUTHYL NITRATE.

BISMUTHI SUBNITRAS.

Chiefly $OBiNO_3.H_2O$=304.

Bismuth, in small pieces	500 Gm
Nitric acid	1,500 ml
Sodium carbonate	2,500 Gm
Ammonia water	1,250 ml
Distilled water, sufficient.	

Mix 800 ml of the nitric acid with 1,000 ml of the distilled water in a capacious glass vessel, add the bismuth, and set aside for twenty-four hours to dissolve. Dilute the solution with 2,500 ml of distilled water, so that the turbidity produced by the addition of the water no longer disappears on stirring; stir well, and again set it aside for twenty-four hours. Then filter.

Dissolve the sodium carbonate in 5,000 ml of distilled water, with the aid of heat, filter the solution, and let it rest until cold. Then add the cold solution of bismuth nitrate very slowly, and with constant, active stirring, to the sodium carbonate solution. Transfer the whole to a wetted muslin strainer, let drain, wash with distilled water until the washings pass tasteless, and then let it drain again as completely as possible.

Put the moist precipitate in a suitable vessel, gradually add the remainder of the nitric acid, being careful to avoid foaming over, and when all has dissolved, also add gradually 1,000 ml of distilled water, and set the solution aside for twenty-four hours. Then filter.

Dilute the filtrate by adding gradually 6,000 ml of distilled water, stirring well. Then add to this, very slowly, the ammonia water, through a small siphon, stirring constantly. Transfer the whole at once to a strainer, and after draining rapidly, pour over the precipitate 8,000 ml of distilled water, let it drain again, and press out as much of the liquid as possible.

Then dry the product on bibulous paper with a gentle heat, and rub it into powder.

Reaction. First the bismuth is dissolved in the nitric acid to form normal bismuth nitrate, thus:

$$2Bi+8HNO_3=2Bi(NO_3)_3+N_2O_2+4H_2O.$$

Then bismuth subcarbonate is made:

$$2Bi(NO_3)_3+3Na_2CO_3.H_2O$$
$$=(BiO)_2CO_3.H_2O+6NaNO_3+2CO_2.$$

The bismuth subcarbonate is next re-dissolved in nitric acid to form normal bismuthous nitrate again:

$$(BiO)_2CO_3.H_2O.+6HNO_3=2Bi(NO_3)_3+4H_2O+CO_2.$$

Finally the normal bismuthous nitrate is converted into subnitrate as follows:

$$Bi(NO_3)_3+2H_3N+2H_2O=OBiNO_3.H_2O+2H_4NNO_3.$$

Notes. The metal dissolves rapidly at first, but toward the last it is best to apply heat if economy of time is desired. Perforated

baskets of stoneware are used for suspending the bismuth in the acid when bismuth preparations are prepared on a moderately large scale, the acid being placed in a stone pot and the basket containing the bismuth suspended just below the surface of the acid. Unless the vessel in which the solution is effected is sufficiently large to obviate all risk and inconvenience from the violence of the reaction, the metal should be added in portions, each portion to be entirely dissolved before more is added. The capacity of the vessel ought to be about three times the combined volume of the acid and water used to dissolve the metal.

The strong solution can not be filtered through paper, for the acid liquid is so corrosive as to destroy the paper, not only defeating the filtration but also discoloring the solution. It is best to filter through coarsely powdered glass or glass wool, or washed asbestos; sometimes the solution is filtered through cotton previously immersed in dilute nitric acid. Decantation is the easiest method, and generally sufficient, especially when large quantities are operated upon. After dilution, however, filtration through paper is entirely practicable.

The object of first preparing subcarbonate of bismuth is to get rid of the arsenic which the metal contained. This arsenic is held in the acid solution as bismuth arsenate, nearly all of which precipitates upon dilution of the liquid with water, and is removed by filtration after standing twenty-four hours. The remainder of the arsenic is left in the last mother-liquor as ammonium arsenate, together with the ammonium nitrate after precipitation of the bismuth subnitrate with ammonia.

The character and composition of subnitrate of bismuth depend greatly upon the method of its preparation. Among the most important influences that affect the character of the product are these: The relative quantity of water used in the precipitation; the length of time the precipitate is exposed to the mother-liquor; the amount and temperature of the water used for the precipitation; the quantity of water and the length of time consumed in washing; the use or omission of ammonia; and the greater or less care bestown upon the various details of the process.

The process given above probably yields the most uniform and satisfactory results. In order to insure success each step of the

process should be carried out in strict accordance with the precise directions given.

Other processes prescribe that the solution of bismuthous nitrate be evaporated to crystallization and the crystals afterward treated with a definite quantity of water. The evaporation must not be carried so far that a cake forms on the bottom of the dish, for then the salt will be hard and difficult to manage. The clear crystals removed from the mother liquor are carefully made dry by gently pressing them between blotting paper, which operation must be very carefully carried out so as not to soil the crystals with fibers from the paper. No heat must be used in drying the crystals. Triturate one part of the crystallized salt very thoroughly with four parts of distilled water, and then pour this mixture into a large vessel containing twenty-one parts of *boiling* distilled water, stirring constantly. Let cool; decant; and wash the precipitate on a strainer with ten parts of cold distilled water, gradually added. After draining as rapidly as possible, press out the last of the wash-water by means of a screw press, and dry the product on filter paper at not over 30° C.

In order to obtain a good product, the nitric acid used must be pure, distilled water must be used, and the acid solutions must not come in contact with wood or other organic substances, or with metals.

Whenever the precipitated bismuth subnitrate is permitted to remain long in contact with the mother liquor in which it has been thrown down, it becomes more dense, and at the same time takes up more nitric acid. This must be avoided. Long contact with much water also renders the preparation heavier, but at the same time more basic. Cold liquids will yield a voluminous precipitate; hot water makes the precipitate more dense. If the water used for precipitating is 50° C. the precipitated subnitrate will have a composition corresponding to about 77 per cent of Bi_2O_3; if the water is boiling hot (100° C.) the subnitrate will have a composition corresponding to about 80 to 81 per cent of Bi_2O_3. According to Loewe, the precipitate is not affected by a solution of ammonium nitrate, and hence it can be washed with such a solution without danger.

The precipitate must be dried at a low temperature (not over 30° C.), but must be thoroughly dry before it is put away. After removing all water that can be gotten rid of by pressing

it, the precipitate should be dried in layers. If not thoroughly dried it decomposes by the influence of light and acquires a nitrous odor. If dried properly in layers, these can be easily broken and powdered by rubbing the pieces through a fine wire sieve by means of a soft brush. If dried at too high a temperature the pieces are hard, not so readily powdered, and apt to yield a coarse, harsh powder.

Good subnitrate of bismuth is soft and bulky.

Description.—A fine, white powder, odorless, almost tasteless; insoluble in water, alcohol, and glycerin.

Bismuth subnitrate is always a comparatively heavy substance, but it must be as bulky as it can be made. The product varies extremely in this particular. A relatively very heavy and coarse preparation is not fit for medicinal uses.

BISMUTHYL CHLORIDE.

BISMUTHI OXYCHLORIDUM.

(Oxychloride of Bismuth.)

$OBiCl=259.4$.

Bismuth	14 Gm
Nitric acid	23 ml
Sodium chloride	39 Gm
Distilled water, sufficient.	

Mix the acid with 30 ml of water; dissolve the metal in the mixture. Dissolve the sodium chloride in 400 ml of distilled water, and filter. Pour the acid solution of bismuth nitrate gradually and with constant stirring into the solution of sodium chloride.

Wash the precipitate with distilled water, first by decantation and afterwards on a filter, until the washings are tasteless. Drain, and then dry the product between bibulous paper with the aid of gentle heat.

Reaction. $OBiNO_3+NaCl=NaNO_3+OBiCl$.

Description.—A fine, white, odorless and tasteless powder.

BISMUTHYL IODIDE.

BISMUTHI OXIODIDUM.

(Oxy-iodide of Bismuth.)

OBiI=350.5.

Bismuth subnitrate	12 parts
Potassium iodide	7 parts
Distilled water	15 parts

Mix and digest together in a bottle for an hour, shaking occasionally. Then transfer to a filter, and wash with warm water until the washings are tasteless. Drain, and dry between filter-paper.

Reaction. $BiONO_3.H_2O+KI=KNO_3+BiOI+H_2O$.

O. Kaspar's Method.

Crystallized normal bismuth nitrate	94.5 Gm
Distilled water	30 liters
Nitric acid, the least quantity sufficient to dissolve the bismuth nitrate in the water.	

Make a solution:

Potassium iodide	33.2 Gm
Distilled water	3 liters
Dissolve.	

Mix the two solutions.

When the brown precipitate has changed to yellowish and finally to brick-red, wash it by decantation and afterward on a filter, and dry it at about 100° C.

B. Fischer's Method.

Crystallized normal bismuth nitrate	95.4 Gm
Glacial acetic acid	120 ml
Potassium iodide	33.2 Gm
Sodium acetate	50 Gm
Distilled water, sufficient.	

Dissolve the bismuth nitrate in the glacial acetic acid. Dissolve the potassium iodide and the sodium acetate in 2 liters of distilled water. Mix the clear solutions, constantly stirring, pouring the bismuth solution gradually into the other.

A yellowish-brown precipitate is formed, which changes to lemon-yellow, and finally to brick-red. Wash the precipitate by decantation and dry it at 100° C.

Should be kept protected from light.

Description.—A brick-red, micro-crystalline powder, odorless, tasteless, insoluble in water and in alcohol.

On ignition it should yield not less than 66 per cent of Bi_2O_3.

BISMUTH TANNATE.

BISMUTHI TANNAS.

Normal bismuthous nitrate	60 Gm
Glycerin	200 ml
Tannic acid	27 Gm

Solution of soda, 240 ml, or sufficient.

Triturate the bismuth nitrate to powder; add the glycerin and stir until dissolved; then gradually add solution of soda until a precipitate no longer forms, stirring well; wash the precipitate with distilled water until the washings are tasteless; drain; add the tannic acid to the moist hydroxide, mix well, and set aside for two hours, stirring frequently. Then transfer the mixture to a filter, wash with distilled water until the washings are tasteless, and dry the product.

Description.—A dirty-white or pale yellowish, odorless and tasteless powder.

BROMINE.

BROMUM.

Br_2=160.

Bromine is a heavy, dark-red, fuming liquid of a peculiar, suffocating odor and its vapor is terribly irritating and dangerous to the eyes and respiratory organs.

Commercial bromine usually contains some chlorine and iodine. It must be kept in small glass-stoppered bottles in a cool place. See also Part II, Vol. I, and Part I of this volume.

Solutions of Bromine.

Water solutions of bromine are frequently required for surgical and other uses. They may be prepared with the aid of potassium bromide. One such solution, which is commonly employed, contains 10 Gm of bromine and 20 Gm of potassium bromide dissolved in one liter of distilled water; another contains 20 Gm of bromine, 10 Gm of potassium bromide and 800 ml of distilled water. But as bromine is soluble in 30 parts of water at 15°, a simple water-solution containing any quantity of bromine below three per cent may be readily made as required.

CADMIUM CARBONATE.

CADMII CARBONAS.

$CdCO_3 = 172$.

Cadmium	10 parts
Nitric acid (68% of HNO_3)	28 parts
Ammonium carbonate	15 parts
Water.	

Granulate the cadmium by pouring the fused metal from a height of about four feet into a vessel of water. Add the granulated metal gradually to the nitric acid previously diluted with about one-half its volume of water. When all of the cadmium has been added and the reaction has subsided, set the solution (with the undissolved residue of metal) on a water-bath and heat until effervescence has entirely ceased. Dilute the solution with about 30 parts of water. Let the whole stand at rest for a few hours. Filter. Put the filtrate in a large vessel and add 400 parts of hot water.

Dissolve the ammonium carbonate in 150 parts of water. Add a small portion of this solution gradually to the solution of cadmium nitrate to precipitate iron from it. When a test portion of the cadmium nitrate solution shows no further test for iron, filter

the liquid and pour it into the remainder of the solution of ammonium carbonate. Wash the precipitated cadmium carbonate by decantation until free from nitrate and ammonium salt, collect it, and dry it.

Reactions. $3Cd+8HNO_3=3Cd(NO_3)_2+4H_2O+2NO.$

then, $Cd(NO_3)_2+(H_4N)_2CO_3=CdCO_3+(H_4N)_2NO_3.$

Description.—An insoluble, amorphous, odorless and tasteless, white powder.

CADMIUM CHLORIDE.

CADMII CHLORIDUM.

$CdCl_2.2H_2O=218.8.$

Saturate hydrochloric acid with cadmium carbonate, digest the solution with a slight excess of the carbonate, filter, and evaporate to dryness. Dissolve the dry salt in two-thirds of its own weight of hot distilled water, acidulate with hydrochloric acid, and set the liquid aside for several hours to crystallize. Dry the crystals with the aid of moderate heat, or in a desiccator over sulphuric acid.

Description.—Colorless crystals, readily soluble in water and in alcohol.

CADMIUM IODIDE.

CADMII IODIDUM.

$CdI_2=365.$

Granulated cadmium	15 parts
Iodine	33 parts
Distilled water	100 parts

Put the metal, iodine and water into a flask and place this in warm water, or keep its contents at about 80°, until the odor of iodine has passed and the metal is nearly all dissolved. Then heat to boiling for a few minutes, filter hot, evaporate to saturation, and set aside to cool and crystallize. Drain the crystals and

dry them between blotting paper, or over sulphuric acid. Evaporate the mother-liquor to recover more crystals.

Reaction. $Cd+I_2=CdI_2$.

Description.—Beautiful, thin, pearly, white plates, odorless, readily water-soluble. Also soluble in alcohol and in ether.

CADMIUM NITRATE.

CADMII NITRAS.

$Cd(NO_3)_2.4H_2O=308$.

Dissolve granulated cadmium in dilute nitric acid to saturation, acidulate the solution, and evaporate to crystallization.

Description.—Colorless, deliquescent, needle-like crystals.

CADMIUM SULPHATE.

CADMII SULPHAS.

$3CdSO_4.8H_2O=976$.

Saturate moderately diluted sulphuric acid with cadmium hydroxide, acidulate the solution with some more of the acid, filter, and evaporate to crystallization.

Notes. The salt may also be made by dissolving granulated cadmium in sulphuric acid of about twenty per cent strength; but the metal dissolves very slowly.

Description.—Large colorless crystals, very readily water-soluble, the solution having an acid reaction.

CALCIUM ACETATE.

CALCII ACETAS.

$Ca(C_2H_3O_2)_2.2H_2O=194$.

Calcium carbonate	3 parts
Acetic acid (36% of $HC_2H_3O_2$)	10 parts
Water	2 parts

Mix in a large porcelain dish and dissolve with the aid of moderate heat. Filter, and evaporate to crystallization.

Description.—White, crystalline, readily soluble in water. Efflorescent. Odorless; taste saline, bitterish.

CALCIUM BENZOATE.

CALCII BENZOAS.

$Ca(C_7H_5O_2)_2.4H_2O=354.$

Lime	1 part
Benzoic acid	4 parts
Water	150 parts

Slake the lime in a porcelain dish with a part of the water. When the reaction has been completed add the remainder of the water, mix thoroughly, and then add the benzoic acid. Boil the mixture a few minutes until the benzoic acid is dissolved. Filter the solution while hot. Evaporate the filtrate to one-half its volume and set it aside to cool. Collect the crystals and drain and dry them. Recover the remainder of the salt from the mother-liquor by evaporation in the usual way.

Notes. Very large, long crystals can be obtained by spontaneous evaporation of a saturated solution. The crystals may be dried with the aid of gentle heat. When the mother-liquor ceases to yield enough to warrant further evaporation, the benzoic acid may be recovered from it by precipitation with a sufficient amount of hydrochloric acid.

Description.—Long, white needles, soluble in about 25 parts of water at 15°, and in a smaller proportion of boiling water.

CALCIUM BROMIDE.

CALCII BROMIDUM.

$CaBr_2=200.$

Ammonium bromide	3 parts
Lime	1 part

Slake the lime with 6 parts of water and triturate it in a mortar, adding enough water to form a liquid mixture. Dissolve the am-

monium bromide in 8 parts of water, heat to the boiling point, and then add the milk of lime until the evolution of ammonia ceases. Then filter the solution and evaporate the filtrate to dryness, stirring constantly so as to produce a granulated salt.

Reaction. $2H_4NBr+CaO=CaBr_2+H_2O+2H_3N$.

Description.—A white, granular salt; odorless; taste sharp, saline. Deliquescent. Soluble at 15° in 0.7 part of water and in 1 part of alcohol; extremely readily soluble in boiling water or alcohol. The solution is neutral to test-paper.

CALCIUM CARBONATE; PRECIPITATED.

CALCII CARBONAS PRAECIPITATUS.

$CaCO_3$=100.

Calcium chloride, fused	10 parts
Sodium carbonate	26 parts
Distilled water, sufficient.	

Dissolve the calcium chloride in 100 parts of the water, and the sodium carbonate in another equal quantity. Filter the solutions. Heat both solutions to about 80° C. Pour the solution of calcium chloride into the solution of sodium carbonate, stirring constantly. Wash the precipitate by decantation; then collect and dry it.

Reaction. $CaCl_2+Na_2CO_3=CaCO_3+2NaCl$.

Notes. Hot solutions are used to insure a dense precipitate, so that the process of washing may be easier; when cold solutions are used the precipitated calcium carbonate is a very bulky magma, difficult to wash free from sodium chloride.

The precipitate should be washed until the washings cease to be rendered turbid by test solution of silver nitrate, and then dried at 100° C.

Another Method.

Prepare a solution of calcium chloride from white marble, as described under the title of Calcium Chloride, removing iron, aluminum and magnesium by employing chlorine water and milk of lime. Render the solution alkaline by using a sufficiency of

milk of lime. Filter. Then acidulate the liquid with hydrochloric acid. Precipitate this solution with either ammonium carbonate or sodium carbonate.

Description.—A very fine, perfectly white powder, odorless, tasteless, and insoluble in water and in alcohol. Under the microscope the precipitated calcium carbonate is seen to be micro-crystalline.

Prepared Chalk.

Triturate 1000 Gm of chalk with a little water until reduced to a very fine powder by the "levigation." Throw the mixture into a vessel capable of holding about twenty liters and nearly filled with water, stir the mixture well, and, after a brief time, decant the supernatant liquid, while yet white and turbid from the suspended particles of the finely divided chalk, into another vessel. To the residue in the first vessel add a fresh portion of water, stir well again, and after allowing the coarser particles to subside as before, decant once more, adding the second turbid liquid to that previously decanted. Set the mixed liquors aside, and when the fine powder has subsided perfectly, pour off the water, and collect and dry the powder.

Note. This process of separating the coarse, gritty particles from the levigated chalk is called "elutriation," the object being to obtain a very soft, finely divided product.

Instead of drying the product as a mass, the moist powder may be formed into cones by "trochiscation."

Description.—A light, amorphous, cream-colored, nearly white, very soft powder, or conical lumps; odorless and tasteless. Insoluble in water and in alcohol.

CALCIUM CHLORIDE.

CALCII CHLORIDUM

$CaCl_2$=110.8.

White marble, in powder	250 Gm
Hydrochloric acid (31.9% of HCl)	500 ml
Water	500 ml
Chlorine water.	
Lime.	

Mix the acid and water. Add the marble in small portions, stirring well, and let the effervescence subside after each addition before adding more. When all the marble has been added and the reaction has nearly ceased, heat the mixture to boiling until no more marble dissolves, replacing the water lost by evaporation. Add to the hot liquid 15 ml of strong chlorine water, stir well, and continue boiling for half an hour. Add milk of lime a sufficient quantity to render the liquid distinctly alkaline. Filter while hot. Evaporate to dryness in a tared dish and fuse the residue at a low red heat.

Keep the product in tightly closed bottles.

Reaction. $CaCO_3+2HCl=CaCl_2+H_2O+CO_2$.

Notes. Marble is liable to contain some iron. Used in excess it reprecipitates dissolved iron and other metals. The chlorine water has for its object the oxidation of the iron to the ferric condition to facilitate precipitation, which is further insured by the addition of milk of lime.

Should the product still contain iron after fusion, it must be redissolved in twice its weight of boiling water, again treated with chlorine water, and also with either milk of lime or pure calcium carbonate, the hot solution filtered, and again evaporated to dryness and the residue fused. In evaporating to dryness add hydrochloric acid occasionally to prevent the product from becoming alkaline.

Description.—Hard white pieces or masses; odorless; taste saline, acrid. Highly deliquescent. Soluble in 1.5 parts of water at 15° and in 8 parts of alcohol; in 1.5 parts of boiling alcohol and freely in boiling water.

CALCIUM HYDROXIDE.

CALCII HYDROXIDUM.

$Ca(OH)_2=74$.

Lime	8 parts
Water	5 parts

Place the lime in a metal pot, pour the water upon it, and when

vapor ceases to be disengaged, cover the pot well and set it aside until the contents are cool. Put the slaked lime into an iron-wire sieve, and by gently shaking cause the fine powder to pass through the sieve, rejecting what is left. Put the powder into a bottle and cork tightly.

Notes. Only recently prepared calcium hydroxide is fit to be used for any of the purposes for which slaked lime is prescribed, as it soon becomes contaminated with carbonate. It should be perfectly white.

The water should be added to the lime all at once; otherwise, when too little water is added at first, the hydrate formed will be coarse and hard, and does not afterward disintegrate readily on addition of more water.

Good, clean, white calcium oxide must be used to obtain a sufficiently pure and white hydroxide.

Calcium Hydroxide Solution.

(LIQUOR CALCIS. LIME WATER.)

A saturated aqueous solution of calcium hydroxide contains about 0.17 per cent of that compound.

Lime	1 part
Distilled water	300 parts
Water, sufficient.	

Slake the lime by the gradual addition of 6 parts of water, then add 300 parts of water and stir occasionally during half an hour. Allow the mixture to settle, decant the liquid and throw this away. Now add to the residue 300 parts of distilled water, stir well, wait a short time for the coarser particles to subside, and pour the liquid, holding the undissolved lime in suspension, into a glass-stoppered bottle. Pour off the clear liquid when wanted for use.

Notes. The object of rejecting the solution first made is to remove dust and alkalies. After this washing the calcium hydroxide furnishes a purer product.

At 19°.5 C. water dissolves about $\frac{1}{800}$ of its weight of calcium hydroxide.

When a solution of calcium hydroxide is exposed to the air,

which happens each time the stopper is removed from the container, some CO_2 from the air precipitates calcium carbonate from the liquid, thereby reducing the quantity of $Ca(OH)_2$ in solution. But if an excess of the hydroxide is allowed to remain in the stock bottle, the solution is kept saturated.

Syrup of Lime.

Lime	65 Gm
Sugar	400 Gm
Water.	

Triturate the lime and sugar thoroughly in a mortar, so as to form a homogeneous powder; then add the mixture to 500 ml of boiling water, contained in a bright copper or tinned iron vessel, boil for five minutes, constantly stirring, and then strain. Dilute the strained liquid with an equal volume of water, and filter through white paper. Then evaporate the filtrate, in a tared capsule, to 700 Gm, allow it to cool, add to it enough water to make the product measure 1000 ml, and mix thoroughly.

Keep the syrup in well-stoppered bottles.

Notes. The preparation contains a chemical compound formed by the lime with the sugar, called calcium sucrate, which may, however, dissolve more lime. It is best to dissolve the sugar in the water, heat to boiling, and then add recently prepared calcium hydroxide in powder. The calcium hydroxide may be prepared by slaking 65 Gm of calcium oxide with 40 Gm of water. The calcium hydroxide may also be dissolved without heat.

Description.—A clear, colorless syrup, of alkaline taste.

CALCIUM HYPOPHOSPHITE.

CALCII HYPOPHOSPHIS.

$Ca(PO_2H_2)_2$=170.

Phosphorus	1 part
Lime	2 parts
Distilled water.	
Carbon dioxide.	

Put the phosphorus in a bottle containing 20 parts of a saturated solution of sodium chloride and large enough to be only about

half filled by these contents. Heat the bottle gradually by placing it in a vessel of hot water (about 50°) until the phosphorus becomes liquid. Shake the bottle until cold. Wash the granulated phosphorus two or three times with distilled water.

Place the lime in a porcelain dish and add 20 parts of water. When the lime has been completely converted into calcium hydroxide and intimately mixed with the water to form a creamy mixture, add the granulated phosphorus and stir well. Heat the mixture at about 40°, stirring frequently, until the phosphorus is all dissolved, which may be known by the fact that the evolution of phosphine ceases.

Now add enough distilled water to make the liquid measure about 40 parts, filter it, and conduct into the filtrate a current of carbon dioxide as long as any precipitate continues to be formed, keeping the liquid at about 60° during this part of the process. Filter out the calcium carbonate.

Evaporate the filtrate over a water-bath to dryness, being careful not to permit the temperature to exceed 60°. Redissolve the dry salt in six times its weight of hot distilled water (not over 60°), filter again (if necessary to make the solution perfectly clear), and evaporate it at 40° in a porcelain dish to about one-half its volume. Let it cool. Collect the crystalline salt which has been formed, drain it well, and dry it with the aid of gentle heat.

Evaporate the mother-liquor to one-half its volume, let it cool, and collect and dry the second crop of crystalline salt in the same manner as the first. Evaporate the second mother liquor in the same way.

Reaction. $3Ca(OH)_2+8P+6H_2O=3Ca(PO_2H_2)_2+2H_3P.$

Notes. When phosphorus is digested with alkaline hydroxides —whether potassium, sodium or calcium hydroxide—the products are hypophosphites and hydrogen phosphide. The reaction is completed when no more phosphine (hydrogen phosphide) is evolved. This gas is known by its strong garlic odor. Owing to its inflammable nature care should be taken that no accident may happen. To guard against danger from a too rapid evolution of phosphine, and from the explosive decomposition of the hypophosphite, the temperature must be kept within safe limits, as directed.

The lime is used in considerable excess. Hence the calcium hydroxide which is contained in the solution with the hypophosphite must be decomposed and the calcium precipitated as carbonate by conducting carbon dioxide into the solution and by heating the liquid to prevent the formation of soluble calcium bicarbonate.

The solution of calcium hypophosphite, filtered free from the precipitated carbonate, is evaporated to dryness, and the salt redissolved in a definite proportion of distilled water. A beautiful product can then be obtained from the filtered solution by crystallization in the manner described.

But in order to obtain a perfect product it is necessary that the lime used shall be pure. It must, therefore, be made by the calcination of a pure calcium carbonate. Distilled water must also be used throughout instead of common natural water.

Description.—Colorless, transparent crystals, or pearly, lustrous scales, or a white crystalline powder; odorless; taste bitter, nauseous. Soluble in 6.8 parts of water at 15°, and in a somewhat smaller quantity of boiling water. Insoluble in alcohol. It must be neutral to litmus paper.

Syrup of Hypophosphites.

Calcium hypophosphite	45 Gm
Potassium hypophosphite	15 Gm
Sodium hypophosphite	15 Gm
Diluted hypophosphorous acid	2 Gm
Sugar	500 Gm
Spirit of lemon	5 ml
Water.	

Triturate the hypophosphites with 450 ml of water until dissolved, add the spirit of lemon and the hypophosphorous acid, and filter the liquid. In the filtrate dissolve the sugar by agitation, without heat, and add enough water, through the filter, to make the product measure 1,000 ml. Strain, if necessary.

Syrup of Hypophosphites with Iron.

Ferrous lactate	10 Gm
Potassium citrate	10 Gm
Syrup of hypophosphites.	

Rub the ferrous lactate and potassium citrate with 50 ml of hot distilled water until dissolved. Filter the solution. Add it to 1,000 ml of syrup of hypophosphites, mix well, and evaporate to 1,000 ml.

This preparation should be freshly made, when wanted.

CALCIUM IODIDE.

CALCII IODIDUM.

CaI_2=293.

Iodine	20 parts
Iron	8 parts
Lime	5 parts
Water, sufficient.	

Digest the iron and 15 parts of the iodine with 30 parts of water, in a flask, until all odor of iodine ceases and a green solution of ferrous iodide is obtained. Filter this, then add the remainder of the iodine, and dissolve.

Slake the lime with 6 parts of water, and triturate the calcium hydroxide with enough water to make a smooth liquid mixture. Add this milk of lime gradually to the solution of iron iodides until the iron has all been precipitated. Then filter the liquid and evaporate it to dryness, stirring constantly.

Reactions.

$Fe+2I=FeI_2$; then $FeI_2+I=FeI_3$; then

$2FeI_3+3Ca(OH)_2=3CaI_2+Fe_2O_3+H_2O$.

Description.—A white, granular salt; odorless; taste acrid, saline, reminding of iodine. Deliquescent. Readily soluble in water and in alcohol.

CALCIUM LACTATE.

CALCII LACTAS.

$Ca(C_3H_5O_3)_2.5H_2O$=308.

Lactic acid (75%)	30 parts
Calcium oxide	7 parts
Distilled water	200 parts

Slake the calcium oxide with about 50 parts of the water; add the remainder of the water to the lactic acid. Then add the milk of lime gradually to the diluted lactic acid in a porcelain dish heated to about 90° C. over a water-bath. Filter the solution while hot. Separate the crystals deposited on cooling, and dry them between blotting paper. Evaporate the mother-liquor to obtain more crystals.

Reaction. $2HC_3H_5O_3+Ca(OH)_2=Ca(C_3H_5O_3)_2.2H_2O$.

Notes. The mother-liquor may be evaporated to dryness to obtain all of the calcium lactate; but this product is granular and not pure.

Description.—Colorless, inodorous crystals, soluble in 9.5 parts of water. Freely soluble in boiling water. Insoluble in alcohol.

CALCIUM OXIDE.

CALCII OXIDUM.

(Calx. Lime.)

$CaO=56$.

Fill a Hessian crucible with pieces of white marble, or with any suitable calcium carbonate, and subject it to a red heat until completely decomposed so that the residue no longer effervesces on being mixed with dilute hydrochloric acid after having been first mixed with its own weight of water.

Keep the product in tightly closed bottles in a dry place.

Reaction. $CaCO_3=CaO+CO_2$.

Description.—Hard, white masses or pieces; odorless; taste sharp, caustic. In contact with air it attracts water and carbon dioxide, and falls to powder. Soluble in 750 parts of water at 15°, and in about 1,300 parts of boiling water. Insoluble in alcohol.

If 8 Gm of CaO be mixed with 5 Gm of water a chemical interaction should at once ensue, and the calcium hydroxide formed should be completely soluble in hydrochloric or nitric acid.

CALCIUM PHOSPHATE.

CALCII PHOSPHAS PRAECIPITATUS.

[Precipitated Tri-Calcium Phosphate.]

$Ca_3(PO_4)_2=310.$

Bone-ash is obtained by the calcination of bones. It is the inorganic residue left on burning bones to whiteness, and consists mainly of tri-calcium phosphate with about ten per cent of calcium carbonate and a little calcium fluoride and magnesium phosphate. It is used in the manufacture of glacial phosphoric acid, precipitated calcium phosphate, sodium phosphate, etc.

It should be white or but slightly grayish, odorless, and almost completely soluble, with but slight effervescence, in dilute hydrochloric acid.

Precipitated tri-calcium phosphate is prepared as follows:

Bone-ash, in fine powder	300 Gm
Hydrochloric acid	450 ml
Ammonia water, sufficient.	

Digest the bone-ash with the acid, diluted with 1,500 ml of water, until dissolved. Boil the solution a few minutes; filter; then add 1,500 ml of boiling water. Now add ammonia water until the mixture acquires an alkaline reaction. Collect the precipitate on a muslin strainer, wash it with hot water until the washings cease to produce a precipitate with test solution of silver nitrate acidulated with nitric acid. Dry the washed precipitate at a temperature not exceeding 100° C.

Reaction. When the crude tri-calcium phosphate is dissolved in hydrochloric acid, the reaction occurring is as follows:

$$Ca_3(PO_4)_2+4HCl=CaH_4(PO_4)_2+2CaCl_2.$$

When ammonia is added to the solution both the acid calcium phosphate and the calcium chloride are decomposed,

$$CaH_4(PO_4)_2+2CaCl_2+4H_4NOH+$$
$$4H_4NCl+4H_2O+Ca_3(PO_4)_2.$$

Notes. Hot liquids are employed for the precipitation in order that the product may be dense and easily washed. If the precipitation be effected in cold water the precipitated calcium phosphate is very voluminous, light, presenting a gelatinous appearance, and the washing becomes difficult.

Description.—A light, white, amorphous powder; odorless and tasteless. Insoluble in water and in alcohol. Assumes a yellowish color when moistened with silver-nitrate test-solution.

CALCIUM-HYDROGEN PHOSPHATE.

CALCII PHOSPHAS PRAECIPITATUS CRYSTALLINUS.

$CaHPO_4.2H_2O=172.$

Calcium chloride	11 parts
Sodium phosphate	36 parts

Dissolve the calcium chloride in 100 parts of distilled water, and the sodium phosphate in 200 parts. Filter each solution. Pour the solution of calcium chloride into the solution of sodium phosphate, gradually and with constant stirring. Wash the precipitate with hot distilled water by decantation and finally on a paper filter, and then dry the product.

Reaction. $Na_2HPO_4+CaCl_2=2NaCl+CaHPO_4$.

Notes. If hot solutions are employed the precipitate is easier to wash because it will then be more compact. Several pharmacopœias contain this calcium phosphate instead of the tricalcium phosphate.

Another Method.

Calcium carbonate	10 parts
Hydrochloric acid	20 parts
Distilled water	65 parts

Make a solution. Add to it a sufficient quantity of ammonia water to produce an alkaline reaction on test-paper. Filter.

Pour this filtered solution of calcium chloride thus prepared into a solution prepared out of:

Sodium phosphate	35 parts
Distilled water	200 parts

Stirring constantly. Decant the mother-liquor. Wash the precipitate by decantation until the acidulated washings no longer produce a precipitate with silver nitrate solution. Dry the product.

Third Method.

Calcium carbonate	20 parts
Hydrochloric acid (32% of HCl)	40 parts
Calcium hydroxide	1 part
Phosphoric acid (85% of H_3PO_4)	3 parts
Sodium phosphate	61 parts
Chlorine water, sufficient.	
Distilled water, sufficient.	

Mix the hydrochloric acid with 60 parts of distilled water, and add the calcium carbonate. When effervescence has ceased heat the mixture until solution is effected.

Let the liquid cool and then add enough chlorine water to impart a distinct odor of chlorine, and mix well. Heat again until the odor of chlorine ceases.

Now add the calcium hydroxide, stir well, and let the mixture stand for half an hour at a temperature of 35° to 40° C.

Filter the solution, add the phosphoric acid to the filtrate, filter again, and let the solution get cold.

Dissolve the sodium phosphate in 300 parts of hot distilled water, filter and let the solution cool to about 20° C.

Pour the solution of calcium chloride gradually and with constant stirring into the solution of sodium phosphate.

Let the mixture stand, stirring frequently, until the precipitate becomes crystalline.

Transfer the precipitate to a cloth strainer and wash it with distilled water until the washings (acidulated with nitric acid) no longer give a precipitate with silver nitrate test-solution.

Let it drain thoroughly; squeeze out as much of the water as practicable, using strong pressure; dry the precipitate with the aid of moderate heat, and reduce it to fine powder.

Description.—A light, white, microcrystalline powder; odorless, tasteless; insoluble in water and in alcohol, but scluble in solution of ammonium citrate.

Syrup of Calcium Lactophosphate.

Precipitated calcium carbonate	25 Gm
Lactic acid	60 ml
Phosphoric acid	36 ml
Orange flower water	25 ml
Sugar	700 Gm
Water.	

To the lactic acid mixed with 100 ml of water, and contained in a capacious mortar, gradually add the calcium carbonate, in portions, until it is dissolved. Then add the phosphoric acid, and triturate until the precipitate at first formed is dissolved. Add 150 ml of water, and filter, rinsing the mortar with 75 ml of water, and passing the rinsings through the filter. To the mixed filtrates add the orange flower water, and, having added the sugar, dissolve it by agitation, without heat, and strain. Lastly, pass enough water through the strainer to make the product measure 1000 ml, and mix thoroughly.

CALCIUM SULPHATE.

CALCII SULPHAS.

$CaSO_4.2H_2O=172.$

Native calcium sulphate or "gypsum" contains two molecules of water. From it the dried calcium sulphate or "plaster" is made.

DRIED CALCIUM SULPHATE.

[Surgical "Plaster of Paris."]

A powder containing about 95 per cent, by weight, of calcium sulphate [$CaSO_4=136$], and about 5 per cent of water; prepared from the purer varieties of native gypsum by carefully heating until about three-fourths of the water has been expelled.

Dried calcium sulphate should be kept in well-closed vessels, carefully protected from moisture.

Notes. The kind of dried calcium sulphate required is one that readily takes up the water necessary for crystallization and

at once sets to a hard mass. An anhydrous calcium sulphate does not do this. Hence when the gypsum is dried the heat must not exceed 105° C., for if that temperature is exceeded the product will be anhydrous and useless. So-called "dental plaster of paris" is well adapted for surgical plaster bandages and casts. It is sold by dealers in dentists' supplies.

Description.—"Plaster of paris" is a fine, white powder, without odor or taste.

From moist air it attracts water, becomes granular, and then loses the property of hardening with water.

When mixed with half of its weight of water, dried calcium sulphate forms a smooth, cohesive paste, which rapidly hardens.

It is soluble in about 410 parts of water at 15° C.; in 388 parts at 38° C., and in 476 parts at 100° C. In alcohol it is insoluble.

CALCIUM SULPHIDE; CRUDE.

CALX SULPHURATA.

Sulphurated Lime.

A mixture containing at least 60 per cent of CaS.

Dried calcium sulphate, in fine powder...	70 parts
Charcoal, in fine powder.....	10 parts
Starch, in fine powder.....	2 parts

Mix the powders thoroughly, pack the mixture lightly into a crucible, cover this loosely, and heat it to bright redness, continuing that heat until the contents cease to have a black color. Let the crucible and contents cool. Reduce the product to powder and at once put it into small glass-stoppered bottles.

Reaction. $CaSO_4+2C=CaS+2CO_2$; and

$CaSO_4+3C=CaS+2CO+CO_2$.

Notes. The starch seems to render the mixture more easily converted by the heat. The reduction is not complete, and the

product always contains calcium sulphate and carbon, amounting to about forty per cent.

Description.—A light gray powder having a faint odor of hydrogen sulphide and a nauseous alkaline taste. Only sparingly soluble in cold water, but freely soluble in boiling water. Insoluble in alcohol. Decomposed by hydrochloric, nitric, or acetic acid with copious evolution of hydrogen sulphide; the undissolved residue consists of calcium sulphate and carbon.

CALCIUM SULPHITE.

CALCII SULPHIS.

$CaSO_3.2H_2O=156.$

Saturate "milk of lime" with sulphur dioxide prepared as described under the title Sulphur Dioxide. Filter the solution and let it stand exposed to the air until the crystals of calcium sulphite have deposited. Collect the crystals and dry them between folds of blotting paper.

Another Method.

Calcium chloride	2 parts
Sodium sulphite	5 parts
Water.	

Dissolve the chloride in 10 parts of water and the sulphite in 50 parts. Mix the solutions. Wash the precipitate with cold water and dry it without heat.

This calcium sulphite contains no water and is amorphous. It may be dissolved in a saturated solution of sulphurous acid, adding a little more of the calcium sulphite to the acid than it is able to dissolve, filtering this solution and setting it aside to crystallize by spontaneous evaporation on exposure to the air.

Description.—White, crystalline powder, very sparingly soluble in water.

The solution of calcium sulphite in sulphurous acid is employed as a preservative of cider, fruit juices, etc.

CALCIUM TETRATHIOSULPHATE SOLUTION.

SOLUTION OF SULPHURATED LIME.

("Vlemingkx's Solution.")

Sublimed sulphur	2 parts
Lime	1 part
Water.	

Add 10 parts of water to the lime and make a uniform mixture of them. Dry and sift the sulphur, add it to the lime mixture, mix well, and then add 25 parts of water. Boil together for one hour, stirring constantly, and replace the water lost by evaporation sufficiently to obtain 10 parts of finished clear solution. Let settle and decant.

Keep the product in well closed bottles.

Notes. The solution contains calcium sulphide and calcium thiosulphate.

The reactions are:

$$CaO+H_2O=Ca(OH)_2;\text{ and}$$

$$3Ca(OH)_2+12S=2CaSS_4+CaSO_3S+3H_2O.$$

It will be found that the theoretical proportions of sulphur and calcium oxide required by these reactions are 8 parts of sulphur and 7 parts of lime. An excess of sulphur is, however, always employed. Normal calcium sulphide is CaS, and a solution of it dissolves large quantities of sulphur, forming, according to many authorities, various compounds. The chief constituent is, however, $CaSS_5$, or calcium tetrathiosulphate.

Description.—A dark red-brown liquid having an odor of hydrogen sulphide.

CARBON.

Purified Animal Charcoal.

The pharmacopœial process for the purification of animal charcoal is as follows:

Put 100 Gm of bone-black into a capacious flask, and add 200 Gm of the official hydrochloric acid and 100 ml of boiling water. Connect the flask with an upright condenser. Apply heat by

means of a sand-bath to the flask and keep the contents boiling gently for about eight hours. Then add 500 ml of boiling water and transfer the mixture to a muslin strainer. When the liquid has run off, return the charcoal to the flask. Add to it 100 ml of hydrochloric acid and the same quantity of boiling water, boil for two hours, and then, again, add 500 ml of boiling water, transfer the mixture to a plain paper filter, and, when the liquid has passed off, wash the residue with boiling water until the washings no longer produce a precipitate but only a slight cloudiness with silver-nitrate test-solution.

Dry the washed charcoal perfectly in a drying oven, and immediately put the product in bottles and close these tightly.

Notes. The hydrochloric acid removes from the crude animal charcoal, or bone-black, all of the calcium phosphate (a little over 80 per cent) and some other inorganic substances, but leaves together with the carbon the siliceous matter (which amounts to about 4 per cent. The product, therefore, is less than 16 per cent of the weight of the bone-black used.

If the bone-black imparts color to solution of KOH when boiled with it, it has not been completely carbonized. In that case it should be treated with that alkali before it is boiled with hydrochloric acid. The bone-black may be freed from animal matter by percolating through it a 2 per cent solution of KOH until the liquid passes colorless, and then water until the percolate becomes tasteless.

When purified animal charcoal has been kept a long time it is no longer effective as a decolorizing agent, but can be renewed by being heated to dull redness in a covered crucible and then allowed to cool.

Description.—A dull-black odorless, tasteless, insoluble powder.

CARBON DIOXIDE.

CARBONEI DIOXIDUM.

[Carbonic Acid Gas.]

CO_2=44.

Put into a flask a convenient quantity of marble or chalk, in small pieces, and add enough distilled water to cover it. Con-

nect this generator by means of the required fittings with a wash-bottle containing distilled water, and provide the wash-bottle with a bent glass tube for conducting the gas wherever it may be necessary. Add hydrochloric acid gradually, through a safety tube, to the marble and water.

Reaction. $CaCO_3+2HCl=CaCl_2+H_2O+CO_2$.

Notes. When calcium carbonate, either in form of marble or chalk, is used for generating carbonic acid gas by double decomposition with hydrochloric acid, the by-product of calcium chloride may be recovered, or its solution may be used for the preparation of precipitated calcium carbonate.

When sulphuric acid is employed instead of HCl, calcium sulphate is formed. Chalk and sulphuric acid are not readily mixed for making carbonic acid in the manufacture of mineral waters on a large scale, owing to the fact that the whiting used contains enough moisture to render it lumpy, so that a portion of it is not acted upon by the acid. Chalk, moreover, contains animal and bituminous matters, and, therefore, yields an impure carbonic acid gas of disagreeable odor and taste, which are with great difficulty removed. Chalk is nevertheless very generally employed, and sulphuric acid is used in preference to hydrochloric acid, because the latter is not only dearer but requires a larger acid chamber. The reaction, when chalk (whiting) and sulphuric acid are the materials used, is:

$$CaCO_3+H_2SO_4=CaSO_4+H_2O+CO_2.$$

The prompt removal of the calcium sulphate from the generator as soon as the chalk has been consumed, is necessary, in view of the fact that a hard mass is soon formed by the residue, which will be found troublesome to remove.

Assuming that the chalk used contains 90 per cent of $CaCO_3$, and the sulphuric acid 92 per cent of H_2SO_4, it will require 10,000 parts of chalk and 9,600 parts of acid to produce 3,960 parts of CO_2. At 20° C. 3,960 Gm of carbonic acid gas measures 2,200 liters.

Sodium bicarbonate furnishes a purer carbonic acid gas than can be obtained from chalk.

Carbonic acid water, or so-called "soda water," is water sat-

urated with carbonic acid under about 50 pounds pressure and at nearly the freezing point. It is now prepared most conveniently from compressed carbon dioxide in liquid form which is manufactured and sold in cylinders.

Water at 0° C. dissolves, under the ordinary atmospheric pressure, 1.8 times its own volume of CO_2. Under the pressure of three atmospheres (45 pounds to the square inch), one volume of water dissolves, at 0° C., 5.4 volumes of CO_2.

CARBON DISULPHIDE.

CARBONEI DISULPHIDUM.

(Bisulphide of Carbon.)

$CS_2=76.$

A clear, colorless, highly refractive, very diffusive liquid, having a strong, characteristic, but not fetid, odor, and a sharp, aromatic taste. Miscible in all proportions with alcohol, ether, chloroform, fixed oils, and volatile oils. Sp. w. 1.268 to 1.269 at 15°. Practically insoluble in water.

It must be kept in tightly stoppered bottles, not more than three-fourths filled, or in tin cans, in a cool place, remote from fire.

CERIUM NITRATE.

CERII NITRAS.

$Ce(NO_3)_3.6H_2O=433.$

Dissolve ceric oxide in dilute nitric acid and evaporate to crystallization. It may also be made from cerous sulphate and barium nitrate.

Description.—Colorless crystals, freely soluble in water and in alcohol.

CERIUM OXALATE.

CERII OXALAS.

$Ce_2(C_2O_4)_3.9H_2O=704.$

Silicate of cerium exists in the minerals cerite and allanite. From these minerals the cerium sulphate is first prepared; then

other salts are made from the sulphate. The oxalate is made from the cerous sulphate and sodium oxalate.

Description.—A white powder, tasteless and insoluble in water, but readily soluble in dilute acids.

CERIUM OXIDE.

CERII OXIDUM.

$CeO_2 = 171$.

Heat cerium oxalate in a Hessian crucible to redness and let the contents cool.

Description.—A reddish powder; insoluble in water.

CERIUM SULPHATE.

CERII SULPHAS.

$Ce_2(SO_4)_3 = 566$.

Ceric oxide is dissolved in sulphuric acid.

The ceric sulphate may be reduced to cerous sulphate by means of sodium thiosulphate.

Description.—Colorless; soluble in water.

CHLORINE.

CHLORUM.

$Cl_2 = 70.8$.

Manganese dioxide	10 parts
Hydrochloric acid	35 parts
Water	25 parts

As a generator use a flask of ample capacity and provided with well fitting perforated rubber stopper carrying the thistle tube, safety tube and delivery tube. Connect the generator in the usual way with a wash-bottle containing water, and having a

delivery tube which may be, in turn, connected with the vessel into which the chlorine gas is to be conducted.

The manganese dioxide must be coarsely powdered and the *fine* powder removed from it. It is put in the generator, all connections are made tight, and then the mixture of acid and water poured into the flask through the thistle tube.

The flask is then heated by means of a Bunsen burner and sand-bath until the evolution of chlorine begins (at about 50° C.). The burner may then be withdrawn to be replaced again only when the reaction seems so slow as to require it.

The chlorine is washed by passing it through water.

Reaction. $MnO_2+4HCl=MnCl_2+H_2O+Cl_2$.

Notes. See Chlorine Solution. The hydrochloric acid may be added gradually as required and further addition discontinued when no further evolution of chlorine is desired. To stop the liberation of chlorine entirely the liquid in the flask is poured off from the remaining manganese dioxide. This liquid, containing manganese chloride, may be reserved for the recovery of the salt from it.

Kipp's apparatus may be used to advantage.

Chlorine Solution.

(CHLORINE WATER.)

A water-solution of chlorine containing not less than 0.4 per cent of that element. This is not a saturated solution, but it is sufficiently strong, and it would be difficult to comply with a requirement of greater chlorine strength because water is decomposed by chlorine, hydrochloric acid being formed, and a stronger solution deteriorates more rapidly. A saturated solution at 15° C. contains about 0.6 per cent of Cl_2.

Apparatus required. A flask of about 500 ml capacity; a sand-bath and Bunsen burner; a Woulff bottle of about one liter's capacity; two glass-stoppered receiving-bottles, each of one liter's capacity; glass tubing, perforated rubber stoppers, rubber tube connections, and safety tubes; and glass-stoppered containers of amber-colored glass and of 200 or at most 250 ml capacity.

The flask to be used as the generator, the Woulff bottle in which

the gas is washed, and one of the receiving-bottles are connected by means of the stoppers and tubing, and provided with the safety tubes.

A vessel of water is also necessary in which the receivers may be placed to cool them with the aid of crushed ice.

Materials. Coarsely powdered manganese dioxide, freed from *fine* powder, 20 Gm; official hydrochloric acid; distilled water which has been boiled a few minutes and allowed to cool again; a sufficient quantity of ice; and some loose cotton.

Process. Put the manganese dioxide (20 Gm) into the flask and place this on the sand-bath.

Put about 120 ml of water into the wash-bottle.

Put 400 ml of water into each receiving-bottle.

Place one of the receiving-bottles in the vessel of water kept at a temperature of about 10° C. by means of the ice, and put into the neck of that bottle some loose cotton around the glass tube through which the chlorine is to enter it.

Now pour into the flask or generator, through the thistle tube, a mixture of 70 ml of hydrochloric acid and 50 ml of water.

Apply gently heat to the sand-bath so as to warm the flask. Increase the heat gradually, so that the liberation of chlorine may proceed rapidly enough but not violently. The chlorine is to be passed through the water in the wash-bottle and then from the upper part of that bottle into the receiver.

When the air in the receiving-bottle shall have been wholly displaced by the greenish-yellow chlorine gas, remove the bottle, close it with its glass stopper, and put the second receiving-bottle (containing, like the first, 400 ml of boiled and cooled water) in its place.

Shake well the receiving bottle just removed so that the chlorine may be dissolved in the water.

Continue the evolution of chlorine and collect the washed gas as before, exchanging the two receiving-bottles, alternately, in the manner described, until the water in both shall have become thoroughly saturated with chlorine.

Then pour the saturated solution into the amber-colored stock-bottles, filling each of them completely, close the filled bottles tightly by their glass stoppers, and keep them in a cool, dark place.

Reaction. $MnO_2+4HCl=MnCl_2+2H_2O+Cl_2$.

Notes. The use of a too finely powdered manganese dioxide would result in a too rapid evolution of gas.

The use of undiluted hydrochloric acid causes the reaction to begin at once without the application of heat; but it would not continue to the end. Experience has taught that a diluted acid, as directed, is preferable. The reaction can be readily controlled by proper regulation of the heat applied.

The temperature of the water in the receiving bottles should not be permitted to fall below 9° C. because the water dissolves the chlorine most readily and in greatest proportion at about 9° to 10°. Should the temperature at which chlorine water is prepared (out-doors) be as low as about 1° to 3° C., or should the water in the receiving bottle be as cold as that, it may cause the formation of crystals of Cl_2+10H_2O. If these crystals are formed in the tube connections these may be closed.

Caution. The operator should not forget that chlorine gas is poisonous when inhaled in such quantities as to prove irritating to the respiratory organs. Hence sufficient precautions must be taken to prevent injury. It is best to make chlorine water under a hood or fume chamber, or out-of-doors.

Should any accident occur whereby the chlorine is caused to escape into the room to such an extent as to cause irritation or danger, the operator should at once neutralize its effects with ammonia. Should any person inhale chlorine, let a little ammonia be *cautiously* inhaled, too, and afterwards a little alcohol or ether.

Preservation. Chlorine water must be kept in comparatively "small bottles" because it does not deteriorate so rapidly when not in contact with air, or, in other words, when the containers are filled. It must be kept in a cool and dark place because a higher temperature would cause the expulsion of the gas from its solution and might even cause a completely filled bottle to burst from the pressure, and light greatly hastens the formation of hydrochloric acid.

But chlorine water should *not* be kept in a place where the temperature is below 4° C.

Description.—A greenish-yellow liquid having the peculiar suffocating odor and disagreeable taste of chlorine. It decolorizes dilute solutions of litmus, indigo and other vegetable coloring matters.

Chlorinated Lime.

(Bleaching Powder. "Chloride of Lime.")

A slightly grayish white, or quite white, granular, *dry* powder, evolving the odor of hypochlorous acid (a somewhat chlorine-like odor). It has a nauseous, acrid, saline taste. Very hygroscopic. Decomposes on exposure to air. Only partially soluble in water. Reacts with alcohol and with glycerin.

Its principal or most important constituent is calcium hypochlorite, $Ca(OCl)_2$ which readily decomposes on the addition of an acid, giving off free chlorine. The chlorine thus liberated is termed the "available chlorine" of the chlorinated lime, and the American Pharmacopœia requires the preparation to contain and produce not less than 35 per cent of available chlorine.

Calcium chloride constitutes a large proportion of the chlorinated lime.

Bleaching powder is produced by the action of chlorine on slaked lime.

Chlorinated lime must be kept in tightly closed vessels, to completely exclude air and moisture, and should be stored in a cool, dry place.

When damp the chlorinated lime should be rejected.

It is largely used as a bleaching agent and as a disinfectant.

To produce chlorine gas for purposes of disinfection, chlorinated lime may be put in a dish, mixed with some diluted acid or vinegar, and placed in the room which is then kept closed for a sufficient period.

CHROMIUM ACETATE.

CHROMII ACETAS.

$Cr(C_2H_3O_2)_3=229.$

Chrome alum	10 parts
Sodium carbonate	9 parts
Acetic acid (36%)	10 parts
Water.	

Dissolve the chrome alum in 40 parts, and the sodium carbonate in 20 parts of water. Add the sodium carbonate solution to the chrome alum, stirring well. Wash the chromium hydroxide with warm water by decantation. Drain well, and dissolve it in the acetic acid with the aid of moderate heat. Evaporate to dryness.

Reaction.

$$2KCr(SO_4)_2+3Na_2CO_3+3H_2O$$
$$=K_2SO_4+3Na_2SO_4+2Cr(OH)_3+3CO_2;$$ and then
$$Cr(OH)_3+3HC_2H_3O_2=Cr(C_2H_3O_2)_3+3H_2O.$$

Description.—A greenish, very soluble salt, extremely difficult to crystallize. The solution when made below 60° is violet, but it becomes greenish when heated.

CHROME ALUM.

ALUMEN CHROMICUM.

$$KCr(SO_4)_2.12H_2O=499.$$

Potassium dichromate	4 parts
Sulphuric acid (92.5% of H_2SO_4)	5 parts
Water	9 parts
Starch	1 part

Put the water in a large enough porcelain dish to hold at least twice the total quantities operated upon. Set the water into rapid rotary motion by stirring with a glass rod. Add the sulphuric acid in a thin stream. Dissolve the potassium dichromate in the mixture. Then add the starch a small quantity at a time, stirring cautiously. When the reaction is nearly ended, heat the liquid, *if necessary*, until it acquires a bluish-green color. Filter the solution and set it in a cold place to crystallize.

Reaction.

$$2K_2Cr_2O_7+8H_2SO_4+3C+40H_2O=4[KCr(SO_4)_2.12H_2O]+3CO_2.$$

Notes. The temperature of the reaction is very high, and the chemical interaction liable to be violent unless the starch be added very slowly. It is necessary that the mixture should remain hot

throughout the whole reaction, but no application of heat from without is necessary, nor should it be resorted to until after the violence of the action has subsided. The liquid must at the end be bluish-green, and not olive-green. Should the bluish-green color be attained without the application of heat, no heating is necessary. Should, on the other hand, the liquid be olive green, heat it cautiously until it acquires a bluish-green color.

In case the crystallization should be much retarded, evaporate the solution somewhat, and then set it in a cold place. Should it still fail to crystallize drop into it a few crystals of chrome alum to start the crystallization.

As the salt is efflorescent it must be kept in tightly closed bottles in a cool place.

Description.—Dark purple, transparent crystals, soluble in 7 parts of water at 15°. The cold solution is reddish-blue, but above 70° it becomes bluish-green.

CHROMIC ANHYDRIDE.

"ACIDUM CHROMICUM."

["Chromic Acid." U. S.]

$CrO_3 = 100$.

Potassium dichromate	3 parts
Sulphuric acid (92.5% of H_2SO_4)	11 parts
Nitric acid (68% of HNO_3).	
Water.	

Put 5 parts of water in a porcelain dish capable of holding about ten times that quantity; set this water into rotary motion by brisk stirring with a glass rod. Add slowly and in a small stream 8.5 parts of the sulphuric acid. Dissolve the coarsely powdered potassium dichromate in the mixture, applying gentle heat to facilitate the solution. Let the liquid stand (at the ordinary room temperature) for about twelve hours in the dish, which should be covered to keep out dust. A mass of crystals of acid potassium sulphate will now be found on the bottom of the dish.

Decant the red liquid from the crystals. Heat it in a large

porcelain dish to a temperature of from 80° to 90°; and then add slowly the remainder (2.5 parts) of the sulphuric acid.

Should any dark-red crystals of chromic anhydride have formed in the liquid, add cautiously just enough water to cause them to be redissolved.

Let the liquid cool and set it aside for twelve hours. Pour off the mother-liquor from the crystals of chromic anhydride. Place the mass of crystals in a funnel and let it be well drained. Then spread the crystals out upon porous tiles and leave them there for an hour, after which transfer them to a funnel again and rinse them a few times with small quantities of cold concentrated nitric acid. Again place the crystalline mass upon fresh porous tiles and let it remain on the tiles for an hour or two. Then drive off the remaining nitric acid adhering to the crystals by heating them in a porcelain dish at from 80° to 90°. Put the product at once in glass-stoppered bottles.

The mother-liquor poured off from the first crop of crystals may be concentrated by evaporation and set aside to deposit an additional crop of crystals of chromic anhydride, which must be drained and otherwise treated in the same manner.

Reaction. $K_2Cr_2O_7+2H_2SO_4=2CrO_3+2KHSO_4+H_2O.$

Notes. It will be seen that the quantity of sulphuric acid prescribed is several times as great as that required according to the equation. This is necessary to cause the chromic anhydride to crystallize out. The crystals can not be drained sufficiently free from sulphuric acid, nor can they be washed with water or recrystallized; but they can be washed with strong nitric acid, which does not affect the chromic anhydride, and the adhering nitric acid can be gotten rid of by evaporation.

Description.—Small, needle-shaped crystals, of a dark-red color, with a suggestion of purplish hue, and of somewhat metallic lustre. Odorless. Deliquescent. Corrosive, and a very powerful oxidizing agent. It is destructive to animal and vegetable tissues, and, therefore, an escharotic. It should not be brought in contact with reducing agents such as tannin, sugar, alcohol, glycerin, etc., as dangerous accidents might occur from the violent reactions liable to take place. Very soluble in water.

Solution of Chromic Acid; B.P.

The British Pharmacopœia contains a formula for a solution of chromic acid. It directs the solution of 1 part of chromic anhydride in 3 parts of distilled water. This solution has the sp. w. 1.185 and its strength is 29.5 per cent expressed in terms of chromic acid (H_2CrO_4), corresponding to 25 per cent of chromic anhydride (CrO_3).

CHROMIUM HYDROXIDE.

CHROMII HYDROXIDUM.

$Cr(OH)_3=103.$

Prepared from chrome alum as described under the head of chromium acetate.

It may also be prepared from chromium sulphate, as follows:

Chromium sulphate	2 parts
Ammonia water (10%)	3 parts
Water.	

Dissolve the chrome alum in an equal weight of water, and add the ammonia. Boil for twenty minutes. Wash and dry the precipitate.

Chromium hydroxide may also be prepared by conducting a strong current of hydrogen sulphide into a solution of potassium dichromate until the precipitate formed becomes green, and then washing and drying it; but this product contains much sulphur.

Description.—A green, insoluble powder.

CHROMIUM SULPHATE.

CHROMII SULPHAS.

$Cr_2(SO_4)_3.15H_2O=662.$

Dissolve 24 parts of dry chromium hydroxide in 25 parts of sulphuric acid of 92.5% strength in a porcelain dish at a temperature of about 40° to 50°. A violet solution is obtained from which crystals are obtained on cooling. The temperature must not be

permitted to exceed 60° at any time, as the solution then becomes green and does not yield any crystals until after standing for several weeks.

It is said that the addition of some dilute alcohol to the purple solution of chromic sulphate facilitates the crystallization of the salt.

Description.—Purple or violet crystals, soluble in less than their own weight of water.

COPPER ACETATE.

CUPRI ACETAS.

$$Cu(C_2H_3O_2)_2.H_2O=199.5.$$

Subacetate of copper	100 Gm
Diluted acetic acid	400 ml
Water	200 ml

Triturate the verdigris with enough water to form a smooth, thin paste. Then add the acid, mix well, transfer the mixture to a porcelain capsule, and heat it over a water-bath at not over 80° C. (176° F.) until solution is effected. Filter while hot. Set aside to cool and crystallize. Collect and dry the crystals on blotting paper in a cool place.

Reaction. $Cu_2O(C_2H_3O_2)_2+2HC_2H_3O_2+H_2O$
$=2(Cu(C_2H_3O_2)_2.H_2O).$

Notes. To obtain good crystals a slight excess of acetic acid must be present during the evaporation and crystallization. Should the liquid become unclear from blue di-basic copper acetate, through loss of acetic acid by evaporation, more acid must be added.

Additional crops of crystals can be obtained from the mother liquor on evaporation.

The total product should be at least equal to the weight of the verdigris used. The crystals should be distinct, deep green, and should make perfectly clear solutions with water, or with ammonia water.

Must be kept in a well corked bottle, and in a cool place.

Second Method.

Copper sulphate	2 parts
Lead acetate	3 parts
Acetic acid, sufficient.	

Dissolve each salt separately in 6 parts of water. If necessary add a little acetic acid to the solution of the lead acetate to render it clear. Filter the solutions. Mix them. Filter. Collect the filtrate containing the copper acetate, evaporate it on a water-bath at not over 80° C. (176° F.) until a pellicle begins to form, taking care to keep acetic acid present in excess. Then set aside to crystallize.

Reaction. $CuSO_4.5H_2O+Pb(C_2H_3O_2)_23H_2O=$
$Cu(C_2H_3O_2)_2.H_2O+PbSO_4+7H_2O$

Third Method.

Copper sulphate	25 parts
Solution of sodium hydroxide (5 per cent)	165 parts
Acetic acid (36 per cent of $HC_2H_3O_2$)	36 parts
Water.	

Dissolve the copper sulphate in 250 parts of water and filter. Pour this solution into the solution of sodium hydroxide, stirring briskly. Wash the precipitated copper hydroxide thoroughly by affusion and decantation of water until the washings are tasteless. Let it drain. Put the moist copper hydroxide in a porcelain dish. Add the acetic acid. Heat gently until solution is effected. Evaporate to crystallization, taking care to keep an excess of acetic acid present in the liquid.

Fourth Method.

Copper acetate may also be prepared by adding a solution of copper sulphate to one of barium acetate until no further precipitation results. Care should be taken to add neither more nor less than is necessary. The liquid must be well stirred and set aside for a day or two before it is filtered and the filtrate evaporated to crystallization.

Reaction. $CuSO_4+Ba(C_2H_3O_2)_2=Cu(C_2H_3O_2)_2+BaSO_4.$

Notes. Handsome rhombic crystals of normal cupric acetate with 5 molecules of water are formed if a solution saturated at 60° C. is rapidly cooled; when warmed to about 30° C. these crystals lose 4 molecules of water and turn green and opaque, but retain their form.

Copper acetate readily loses acetic acid and water, becoming basic, insoluble and blue or light green. Hence the necessity of keeping the salt in a tightly closed bottle in a cool place.

In the process of preparation it should not be exposed to the air and to heat any more than is unavoidable.

Description.—Dark green crystals with metallic taste and acetous odor. Completely soluble in water, and difficultly soluble in alcohol.

COPPER CHLORIDE.

CUPRI CHLORIDUM.

$$CuCl_2.2H_2O=170.3.$$

Saturate hydrochloric acid with freshly precipitated and well washed copper subcarbonate (made from cupric sulphate with sodium carbonate). Evaporate the solution to the density of about 1.44 and set it in a cool place to crystallize.

It may also be made from cupric sulphate and barium chloride by metathesis.

Description.—Slender, green, deliquescent crystals, soluble in less than their own weight of water.

COPPER NITRATE.

CUPRI NITRAS.

$$Cu(NO_3)_2.3H_2O=241.$$

Copper sulphate	25 parts
Barium nitrate	26 parts

Dissolve the copper sulphate in 200 parts of water and the barium nitrate in 300 parts of water. Filter the solutions. Pour the solution of barium nitrate into the solution of copper sulphate,

stirring well. Reject the precipitated barium sulphate. Filter the solution of copper nitrate, acidify it with nitric acid, and evaporate it to obtain the product in crystals.

Redissolve the crystals in distilled water, acidify with nitric acid, and recrystallize.

Reaction. $CuSO_4.5H_2O+Ba(NO_3)_2=Cu(NO_3)_2.3H_2O+BaSO_4 2H_2O.$

Another Method.

Copper nitrate may also be made by saturating diluted nitric acid with metallic copper, and crystallizing at about 25° C.

Reaction. $3Cu+8HNO_3=3Cu(NO_3)_2+4H_2O+2NO.$

Description.—Beautiful, transparent, deep-blue crystals, readily soluble in water.

COPPER OLEATE.

CUPRI OLEAS.

$Cu(C_{18}H_{33}O_2)_2=625.5.$

Copper sulphate	60 Gm
White castile soap, in fine powder.....	150 Gm

Dissolve the copper sulphate in five liters of water, and the soap in three liters of hot water. Pour the soap solution into that of the copper sulphate, stirring well. Wash the precipitated oleate twice, using ten liters of hot water each time. Collect the dark-green oleate, squeeze the water out of it, and fuse it by very gentle heat, over the water-bath.

Reaction. $CuSO_4+2NaC_{18}H_{33}O_2=Cu(C_{18}H_{33}O_2)_2+Na_2SO_4.$

Notes. The product is about 290 Gm of a dark-green, waxy plaster, containing 12.67 per cent of copper oxide.

COPPER OXIDE.

CUPRI OXIDUM.

$CuO=79.5$.

Heat copper nitrate strongly until no more red vapors pass off and the residue is a fine, black powder.

$2Cu(NO_3)_2=2CuO+2N_2O_4+O_2$.

Cupric oxide is insoluble in water, but readily soluble in dilute hydrochloric, nitric, sulphuric and acetic acids.

COPPER SUBCARBONATE.

CUPRI SUBCARBONAS.

Cupric sulphate	5 parts
Sodium carbonate	6 parts
Water.	

Dissolve the blue vitriol in 20 parts of hot water and filter. Dissolve the sodium carbonate also in 20 parts of water and filter. Add the hot copper solution slowly or in small portions to the hot sodium carbonate solution in a large vessel, being careful not to let the liquid run over from the active effervescence. Wash the precipitate with hot water by decantation and then dry it.

Description.—A green powder having a variable composition. Insoluble in water but readily soluble in acetic, sulphuric, hydrochloric and nitric acids.

COPPER SULPHATE.

CUPRI SULPHAS.

$CuSO_4.5H_2O=249.5$.

Copper, in filings	5 parts
Sulphuric acid	8 parts
Nitric acid	4 parts
Water	30 parts

Mix the ingredients in a flask and heat at first gently, and afterwards raise the temperature gradually to the boiling point. Boil until the evolution of gas ceases, evaporate the solution to dryness, dissolve the residue in four times its weight of water, filter and evaporate to crystallization. Dry the crystals without the aid of heat.

Reaction. $3Cu_2+6H_2SO_4+4HNO_3=6CuSO_4+2N_2O_2+8H_2O$.

Granulated copper sulphate. Sulphate of copper, reduced to coarse powder, is dissolved in an equal weight of boiling water, and the solution filtered and evaporated during constant stirring until nearly dry, after which the residue is dried by spreading it in thin layers on paper and exposing it to currents of dry air.

Precipitated copper sulphate may be made by dissolving the salt in its own weight of boiling water, and filtering the solution into an equal volume of alcohol.

Turbidated copper sulphate may be made by dissolving 20 parts of coarsely powdered copper sulphate in 20 parts of boiling water, acidifying with 1 part of diluted sulphuric acid, filtering the solution while hot, and stirring the filtrate briskly until cold. The granular salt thus obtained is collected on a filter, drained, dried at the ordinary temperature, and bottled. The mother liquor is evaporated to one-half its weight and an additional amount of product obtained from it in the same manner. All of the remaining salt may be recovered by evaporation nearly to dryness, and finishing the drying by exposure to the air.

Purification of Commercial Blue Vitriol may be effected as follows:

Put the bluestone or blue vitriol into a porcelain dish. Add 3 parts of water. Apply heat and bring the solution to the boiling point. Then add small quantities at a time of nitric acid, stir well, and continue heating. When the liquid no longer gives off nitrous vapors on the further addition of nitric acid, boil it a few minutes longer, and then let it cool. Add to the cold solution enough water of ammonia to impart a permanent ammoniacal odor to it. Let it stand a few hours. Filter. Evaporate to dryness, and heat the residue strongly until it becomes nearly white. Then let it cool, dissolve it in 2.5 parts of water, filter the solution, add 0.1

part of diluted sulphuric acid, mix well, and set the liquid aside to crystallize by the spontaneous evaporation of the water.

Collect, drain and dry the crystals, avoiding loss of any of their water of crystallization.

Iron is removed by this process.

Description.—Copper sulphate consists of large, transparent, clear, deep-blue crystals; or a blue, granular, crystalline, coarse powder if granulated, precipitated by alcohol, or turbidated. It is odorless, and has a nauseous, strongly metallic taste. Slowly loses its water of crystallization in dry air.

Soluble, at 15° C., in about 2.6 parts of water, and in 0.5 part of boiling water; almost insoluble in alcohol.

When carefully and continuously heated to 30° C., the salt loses 2 of its 5 molecules of water (14.43 per cent), and is converted into a pale-blue, amorphous powder. Two more molecules of water are lost at 100° C., while the fifth is retained until 200° C. is reached, when a white, anhydrous powder remains (63.9 per cent of the original weight). At a still higher temperature sulphur dioxide and oxygen are given off, and a residue of black cupric oxide is left.

The aqueous solution (1 in 20) has a blue color and shows an acid reaction on litmus paper.

AMMONIATED COPPER.

CUPRUM AMMONIATUM.

Copper sulphate	3 parts
Ammonium carbonate	4 parts

Crush the salts; triturate them together in a porcelain mortar.

Notes. The reaction between the salts causes the mixture to become wetted by the water of crystallization liberated from the decomposed copper sulphate. The mixture acquires a deep-blue color. An "ammoniacal" copper sulphate is formed, the composition of which is uncertain. Effervescence takes place from the separation of CO_2.

When effervescence has ceased and the mixture has been re-

duced to a dry powder this product is to be kept in a tightly stoppered bottle.

This preparation is nearly similar in composition to the crystalline ammoniacal copper sulphate, which is more uniform, of handsome appearance, and prepared as follows:

Ammoniacal Copper Sulphate.

Copper sulphate, in powder	10 Gm
Ammonia water	30 ml
Alcohol of 85% strength	60 ml

Dissolve the sulphate of copper in the ammonia water contained in a porcelain capsule, without applying heat. (Heat is generated by the chemical solution.) When the liquid has become cold, filter it. Put the alcohol in a tall, narrow cylinder. Then run in under the alcohol 10 ml of water by means of a funnel tube reaching to the bottom of the cylinder. Finally add the copper solution through the same tube so that it may form a separate layer under the water. Set it aside, without disturbing it, and well corked, for one to four weeks, and then decant the mother liquor, and throw it away. Collect the crystals and dry them as rapidly as possible without the aid of heat by gently pressing them between bibulous paper, avoiding exposure to the air as far as practicable. Put the product at once into a bottle and cork it tightly.

Description.—The preparation is in handsome, dark-blue, needle-like crystals, having an ammoniacal odor and a nauseous, metallic taste. Soluble in 1.5 parts of water.

It is not "ammonio-copper sulphate," which may be made by mixing solutions of the sulphates of ammonium and of copper.

Ammoniacal copper sulphate readily absorbs oxygen from the air, and hence acts as an oxidizing agent on substances present in its solution. Thus, sodium thiosulphate is rapidly converted into sulphate; asparagin and glycocoll to oxalic and carbonic acids, etc.

Copper Potassium Sulphate.

Copper sulphate	100 parts
Potassium sulphate	70 parts
Concentrated sulphuric acid	12 parts
Water.	

Dissolve the copper sulphate in 150 parts of hot water, and the potassium sulphate in a similar quantity of hot water, to which the sulphuric acid has been added. Mix the solutions. Let the mixture stand until cold. Collect the light-blue crystals and dry them. Evaporate the mother-liquor to one-half and let cool again to obtain a second crop.

Aluminated Copper.

(Lapis Divinus. Cuprum Aluminatum.)

Copper sulphate	15 parts
Alum	15 parts
Potassium nitrate	15 parts
Camphor	1 part

Powder the salts and mix them. Fuse the mixed powder in a porcelain dish, stirring well. Let the mass cool. Powder it. Finally add the camphor and mix the whole intimately by trituration.

GOLD; PURE.

AURUM PURUM.

Au=197.

Gold, in coin or scraps	12 parts
Hydrochloric acid (38.67% of HCl)	32 parts
Nitric acid (68% of HNO_3)	7 parts
Ferrous sulphate	55 parts
Distilled water.	

Put the gold in a flask with the hydrochloric acid. Apply gentle heat. Add the nitric acid, a little at a time, until the metal is completely dissolved (leaving only the precipitated silver chloride formed by the silver contained in the gold coin or scraps, if alloyed with silver). Filter the solution through a paper filter, washing the silver chloride on the filter with a little distilled water and adding the washings to the filtrate. Evaporate the filtrate to a syrupy consistence, weigh it, and dilute it with ten times its weight of distilled water.

Dissolve the ferrous sulphate in 250 parts of distilled water,

acidulate the solution with 5 parts of diluted hydrochloric acid, heat to boiling, filter, and let the filtrate cool.

Add the solution of ferrous sulphate very gradually and with constant stirring to the solution of gold chloride. Set the mixture in a warm place for an hour. Then collect the yellowish-brown precipitate of metallic gold upon a paper filter, wash it first with a little diluted hydrochloric and afterwards with distilled water. Dry the product with the aid of moderate heat.

Reactions. First, $2Au+3Cl_2=2AuCl_3$; then, $2AuCl_3+6FeSO_4=2Au+2Fe_2(SO_4)_3+Fe_2Cl_6$.

Notes. Coin gold and gold employed in making jewelry and gold vessels is alloyed with either silver or copper, or both. The silver forms insoluble silver chloride when the gold coin is dissolved in the hydrochloric and nitric acids. [This silver chloride should be collected and may be reduced to metallic silver, when convenient, as described under the title of Silver; Pure] Copper dissolves together with the gold, but remains in the liquid when the gold is precipitated with ferrous sulphate.

The solution of gold chloride and that of ferrous sulphate should be perfectly clear when about to be mixed. Ferrous chloride may be used in place of ferrous sulphate.

The precipitated gold is chemically pure and so finely divided that it is easily dissolved in hydrochloric and nitric acids mixed together.

Oxalic acid (pure) may be used instead of ferrous sulphate for the precipitation of the gold.

$$2AuCl_3+3H_2C_2O_4=2Au+6HCl+6CO_2.$$

The amount of crystallized oxalic acid required for 12 parts of gold is about 12.5 parts.

GOLD CHLORIDE.

AURI CHLORIDUM.

$AuCl_3=302.8$.

Pure gold	10 parts
Hydrochloric acid (38.67% HCl)	26 parts
Nitric acid (68% HNO_3)	7 parts

Mix the acids. Dissolve the gold in the mixture with the aid of gentle heat. Evaporate the solution to dryness. Fuse it at a temperature not exceeding 150°.

Put the dry salt in a perfectly dry glass-stoppered bottle and keep it in a cool, dark place.

Notes. The amount of nitric acid prescribed here is probably just sufficient. More is usually ordered. An excess should not be used. Gold chloride is unstable; it keeps better if mixed with sodium chloride. The pharmacopœias generally prescribe a mixture of equal parts of gold chloride and sodium chloride, but some pharmacopœias prescribe a smaller proportion of the sodium chloride.

Description.—Golden yellow, transparent crystals; odorless; taste metallic. Deliquescent in moist air.

Chloride of Gold with Sodium Chloride.

Pure gold	10 parts
Pure sodium chloride	15.4 parts
Hydrochloric acid of 38.67% strength	26 parts
Nitric acid of 68% to 70% strength,	sufficient.

Mix the hydrochloric acid with 6 parts of the nitric acid. Add the gold. Let the gold dissolve, adding more nitric acid later on, if necessary, to effect the solution of the gold, but taking care not to add more than is necessary. Apply gentle heat to facilitate the solution. When all of the gold has been dissolved evaporate the solution to dryness and fuse the residue at as low a temperature as possible, not exceeding 150°. Then redissolve the gold chloride in 30 parts of distilled water, dissolve the sodium chloride in the gold solution, then evaporate to dryness, and reduce the product to coarse powder. Keep it in a glass-stoppered bottle.

Keep the product in a dark place.

Notes. The gold chloride obtained by dissolving gold in nitrohydrochloric acid and evaporating the solution to dryness without subsequently fusing the salt is a mixture of aurochloric acid ($HAuCl_4.2H_2O$) and gold trichloride. The unusually strong hydrochloric acid ordered in these formulas is preferable to the official acid. A hydrochloric acid of 38.67% strength has the sp. w. of about 1.19.

The pure gold required is precipitated as described under Gold.

Description.—The gold and sodium chloride of the American Pharmacopœia consists of equal weights of gold trichloride and sodium chloride. It is a coarse, orange yellow, granular powder; odorless; taste saline, metallic. It is slightly deliquescent in moist air. Readily soluble in water. Alcohol dissolves the gold chloride, leaving the sodium chloride, which should constitute not more than one-half by weight of the whole quantity added to the alcohol.

Another Method.

This preparation may be made as follows:

Pure gold	13	parts
Hydrochloric acid (32%)	37.5	parts
Nitric acid (68%)	6	parts
Distilled water	40	parts
Dry pure sodium chloride	20	parts

Dissolve the gold in the mixed acids. Add the water. Dissolve the sodium chloride in the liquid. Evaporate the liquid over a water-bath to dryness.

Notes. In this formula, which is constructed after that of the German pharmacopœia, there is rather more hydrochloric acid and less nitric acid prescribed than in other working formulas.

Another Method.

(After the Swiss Pharmacopœia.)

Pure gold	65	parts
Hydrochloric acid (25% of HCl)	180	parts
Nitric acid (32.3% of HNO_3, or 1.2 sp. w)	60	parts
Sodium chloride	100	parts
Distilled water	100	parts

Mix the acids. Dissolve the gold in the mixture with the aid of gentle heat. Evaporate the solution over a water-bath to the consistence of syrup, so that upon cooling it forms a solid salt mass. Dissolve this gold chloride in the distilled water, add the completely dried (decrepitated) sodium chloride, and evaporate to dryness over a water-bath.

HYDROGEN.

$H_2=2$.

Hydrogen gas, when required for laboratory operations, may be made as follows:

Zinc granulated	70 parts
Sulphuric acid	100 parts
Water	500 parts

Put the zinc in a wide-mouthed half-gallon bottle provided with a twice perforated rubber stopper, fitted with thistle-tube and a bent delivery tube. Connect the delivery tube with a wash-bottle of about a quart's capacity containing a pint of water, and provide the wash bottle with the necessary delivery tube, which may be, in turn, connected with any bottle or tube into which the hydrogen is to be conducted.

Add the sulphuric acid to the zinc through the thistle tube, as required.

The most convenient apparatus for producing hydrogen, as well as hydrogen sulphide and several other gases, is Kipp's apparatus.

Reaction. $Zn+H_2SO_4=ZnSO_4+H_2$.

Sixty-five Gm of zinc, if all consumed, will furnish 2 Gm of hydrogen according to this equation. One cubic-decimeter of hydrogen at 0° C., bar. 760 mm., weighs about 0.09 Gm, and 2 Gm of hydrogen, therefore, occupies about 11.16 cubic-decimeters.

HYDROGEN DIOXIDE SOLUTION.

LIQUOR HYDROGENII DIOXIDI.

A water-solution containing, when fresh, about 3 per cent of hydrogen dioxide, H_2O_2, corresponding to about 10 volume units of free oxygen obtainable from each volume unit of the solution.

Apparatus required for the preparation of about 1 liter: Two half-gallon bottles. White paper filters of 30 Cm diameter and of such character as to admit of rapid filtration. A funnel to correspond to the size of the filter.

Materials. Barium dioxide, 300 Gm; phosphoric acid (85%), about 100 ml; diluted sulphuric acid, about 2 ml; starch, in powder, about 10 Gm; distilled water, about 1 liter.

Process. Put 500 ml of cold distilled water into a half-gallon bottle. Add gradually the coarse powder of the barium dioxide, and shake after each addition to break up any lumps that might be formed. When all of the barium dioxide has been added shake the bottle vigorously so that the contents may form a uniform mixture.

Place the bottle in a vessel of water cooled by additions of broken ice so that the contents may be kept at a temperature somewhat below 10° C. (50° F.) for about half an hour. During that time shake the bottle thoroughly every few minutes. Continue keeping the mixture cool and shaking it occasionally and strongly until the barium dioxide shall have been converted into hydroxide, which may be known by the fact that but little water is separated on standing and that a uniform mixture may be obtained, without difficulty, by shaking.

Put into the other half-gallon bottle 96 ml of phosphoric acid and 320 ml of distilled water, and cool this mixture to about 10° C. Take of this liquid 50 ml and set that aside. To the remainder of the mixture of phosphoric acid and water add, in four equal portions, the well-mixed magma from the first bottle, shaking the mixture vigorously and cooling it after each addition. Note the reaction of the mixture upon litmus paper, and, when it shows an alkaline reaction, add cautiously enough of the reserved dilute phosphoric acid to render the reaction again acid.

Repeat the vigorous shaking of the bottle and the gradual addition of more phosphoric acid until the liquid no longer becomes alkaline upon long continued strong shaking. (Should the reserved portion of the mixture of phosphoric acid and water prove insufficient to effect a slightly acid reaction, mix a sufficient additional amount of phosphoric acid and distilled water in the same proportions as before, and add of this as much as may be necessary.)

When the whole mixture shall have been finally thoroughly shaken until the liquid portion has acquired a neutral reaction on test paper, set the bottle aside and let it rest until the supernatant liquid occupies about two-thirds of the volume of the whole mix-

ture. Then decant that liquid upon a wetted double filter of white filter paper about 30 Cm in diameter. When the liquid has passed through the filter, transfer the thick mixture containing the precipitate to the same filter. Rinse the bottle with 100 ml of distilled water and pour this also upon the filter. Wash the precipitate on the filter with more distilled water collecting the washings in the previous filtrate until the total quantity of filtered liquid measures 1 liter.

Add to this filtrate 20 drops of diluted sulphuric acid and shake well. Filter a small portion of the liquid, and add to this clear sample a few drops of diluted sulphuric acid again. Should this render the clear solution cloudy, continue to add small quantities of diluted sulphuric acid until a further addition of it no longer produces cloudiness in a filtered sample. (The filtration of the test samples may be rendered more effective by the addition of a little starch.)

Add to the whole liquid about 10 Gm of starch and shake the mixture thoroughly. Filter the liquid through a wetted white paper filter of the same size as before, returning to the filter the first portions of the filtrate, until a clear liquid passes through.

Assay the filtrate by the method given below and dilute it with distilled water so that the final product shall contain 3 per cent of absolute hydrogen dioxide.

Assay process. Dilute 10 ml of the solution with 90 ml of distilled water. Put 17 ml of the liquid in a beaker, add 5 ml of diluted sulphuric acid, and then run into the mixture, from a burette, a sufficient quantity of decinormal volumetric solution of potassium permanganate to impart a faint pink tint which does not at once disappear on stirring. Each ml of the volumetric solution required corresponds to 0.0017 Gm of H_2O_2.

The number of ml of permanganate solution decolorized by each 1.7 ml of the solution of hydrogen dioxide multiplied by 0.33 gives a product expressing the number of volumes of oxygen yielded by one volume of its solution.

The number of ml of permanganate solution decolorized by each 1.7 ml of solution of hydrogen dioxide divided by 10 gives a quotient expressing the per cent by weight of H_2O_2 contained in the solution.

Instead of taking 1.7 ml of the solution for the valuation (17

ml of the diluted solution), 1 ml may be taken (using 10 ml of a mixture made of 10 ml of the solution and 90 ml of distilled water). The number of ml of permanganate solution decolorized by each ml of the solution of hydrogen dioxide when multiplied by 0.56 gives a product expressing the number of volumes of oxygen yielded by one volume of the solution of H_2O_2. The number of ml of permanganate solution decolorized by each ml of solution of hydrogen dioxide when multiplied by 0.17 gives a product expressing the per cent of H_2O_2 contained in the preparation. Each 1.7 ml of the solution of H_2O_2 should require not less than 30 ml of the volumetric permanganate solution, or each ml should require not less than 17.65 ml. The reaction which takes place in the assay process is:

$$5H_2O_2+3H_2SO_4+2KMnO_4=K_2SO_4+2MnSO_4+8H_2O+5O_2.$$

Thus 2 molecules of $KMnO_4$ are required for 5 molecules of H_2O_2. As the mol. weight of $KMnO_4$ is 158 and that of H_2O_2 is 34, and as the permanganate solution is a decinormal one it follows that each ml of the volumetric solution, containing 0.00316 Gm of $KMnO_4$ corresponds to 0.0017 Gm of H_2O_2 or to 0.0008 Gm of *available* O_2.

Notes. The first reaction by which the barium dioxide is decomposed is:

$$BaO_2+2H_2O=Ba(OH)_2+H_2O_2.$$

The completion of this reaction is facilitated by cooling the water below 10° C., and may be known to have been attained when a magma has been formed from which but little water separates on standing.

The barium is then precipitated by means of phosphoric acid.

$$3Ba(OH)_2+2H_3PO_4=Ba_3(PO_4)_2+6H_2O.$$

If necessary a little sulphuric acid may be used, as directed by the Pharmacopœia, to complete the separation of the barium.

All that then remains to be done is the filtration of the liquid and the adjustment of its strength to the prescribed standard.

The filtration is frequently difficult. A double filter is necessary and starch is added to further aid the clarification.

Small quantities of phosphoric and sulphuric acids are unavoidably left in the product, and their presence tends to preserve it.

Hydrogen dioxide is very unstable, and its uses depend upon that fact; it decomposes into water and oxygen. Heat and light hasten this decomposition. Hence it must be kept in a cool place and protected from light. But it can not be kept in tightly stoppered bottles because the containers would burst should the dioxide decompose in them.

Solution of hydrogen dioxide may be evaporated on a water-bath, at a temperature not exceeding 60° C., until reduced to one-fifth of its original volume, being thus rendered five times the official strength, or made of such strength that each volume of the concentrated solution will yield fifty volumes of oxygen upon decomposition of the hydrogen dioxide it contains.

One per cent of boroglycerin is said to retard the decomposition of the solution. It should be kept in a cool, dark place.

Description.—A colorless, odorless liquid, slightly acidulous in taste and producing a peculiar sensation and soapy froth in the mouth. Reaction acid, owing to the free acid left in the liquid to preserve it. The dioxide is liable to decomposition when the solution is rapidly heated to a temperature above 60° C.

HYDROGEN SULPHIDE.

$H_2S=34$.

When hydrogen sulphide is required for laboratory operations, as in the preparation of hydrobromic or hydriodic acid, it may be produced as follows:

Put any convenient and suitable quantity of ferrous sulphide in small fragments into a Kipp's apparatus (Fig. —) and add diluted sulphuric acid, or diluted hydrochloric acid, as required. The ferrous sulphide is first covered with water in the apparatus, and the dilute acid then added as may be necessary.

IODINE.

IODUM.

I=126.5.

Commercial iodine is easily resublimed. Small quantities may be resublimed in a dish heated in a sand-bath the sublimate being collected in an inverted funnel which is placed over the contents of the dish.

Resublimed or purified iodine consists of dry, brittle, shining purplish-black crystals of a strong characteristic odor and acrid taste. Soluble in alcohol.

Iodine imparts yellowish-brown or deep brown stains to the skin and to articles with which it comes in contact. The stains and odor are persistent. Mortars and other apparatus stained with iodine may be cleaned with potassium or sodium hydroxide, ammonia, potassium iodide or sodium thiosulphate; but the stains on the hands and organic substances generally can not be thus removed.

Iodine must be kept in glass-stoppered bottles.

Compound Iodine Solution.

(LUGOL'S SOLUTION.)

Dissolve 5 Gm of iodine and 10 Gm of potassium iodide in 85 Gm of distilled water.

Keep the solution in a glass-stoppered bottle.

Description.—A deep-red solution of strong iodine odor.

Tincture of Iodine, U. S. P.

Reduce 70 Gm of iodine to coarse powder by trituration in a mortar. Transfer it to a graduated glass-stoppered bottle of about one and one-half liters' capacity. Rinse the mortar with several successive portions of alcohol, and pour these washings into the bottle. Add enough additional alcohol to make the total volume of the contents of the bottle measure one liter. Set the bottle in a warm place. Agitate it occasionally until the iodine is dissolved.

Notes. The official tincture of iodine is so nearly a saturated solution at ordinary low room temperature that it would be unwise to increase its strength to 10 per cent, which is the standard of some pharmacopoeias. The solution takes place so slowly that it is best to powder the iodine and to put the mixture in a warm place to reduce the time required for its completion.

Description.—A deep red solution of strong iodine odor. But the peculiar odor of *an old tincture* of iodine indicates that it contains not only iodine and alcohol but also small quantities of some products formed by interaction between them.

Decolorized Tincture of Iodine.

Iodine	4 Gm
Alcohol	40 ml
Stronger water of ammonia	9 ml

Powder the iodine by trituration. Dissolve it in the alcohol. Add the ammonia water carefully without shaking or stirring the mixture.

Let the mixture stand exposed to sunlight until decolorized, which will require from a few days to several weeks.

Notes. As explosive nitrogen iodide is liable to be produced when the ammonia is added to the iodine solution, any dark-colored sediment formed in the liquid should warn the operator to leave the mixture at perfect rest until the precipitate has disappeared, which it will finally do. If a few drops of phenol be added to the mixture it becomes decolorized at once on shaking.

The preparation contains not iodine, but ammonium iodide and ethyl iodide.

Another Formula.

Iodine	10 parts
Sodium thiosulphate	10 parts
Ammonia water	15 parts
Alcohol	75 parts

Distilled water, sufficient.

Dissolve the iodine and the sodium thiosulphate in 10 parts of distilled water; add the ammonia water, and, finally, the alcohol. Let the mixture stand three days, and then filter.

Notes. This preparation contains sodium iodide, ammonium iodide, ethyl iodide, sodium tetrathionate, alcohol and water.

IRON.

FERRUM.

Powdered Iron.

Metallic iron prepared by filing the metal and triturating the filings in steel mortars. The iron used for this purpose may be either cast iron, wrought iron, or steel. Wrought iron is, however, extremely difficult to powder, and cast iron contains rather too much carbon.

Description and Tests.—A heavy, gray, impalpable powder.

Soluble in dilute hydrochloric acid without residue except traces of carbon. The gas evolved when it is dissolved in HCl should have no odor of H_2S (absence of *sulphur*). The solution oxidized with nitric acid and completely precipitated with ammonia should not yield a blue liquid (absence of *copper*). It should be free from arsenic.

Uses. Powdered iron ("ferrum pulveratum") is used in medicine in the same way as "reduced iron."

Reduced Iron.

Prepared by reducing basic ferric hydroxide or ferric oxide to metallic iron by means of hydrogen, at a sufficiently high heat.

Ferric hydroxide is prepared by precipitation from a solution of ferric sulphate by means of ammonia in the usual way, and the thoroughly washed precipitate is dried. This dried ferric hydroxide is introduced into an iron tube and confined to the middle of that tube by means of asbestos plugs. The tube is placed in a furnace and the middle portion of it, containing the basic ferric hydroxide, is heated to "a strong but not bright red" heat. Then hydrogen, obtained by the action of zinc on sulphuric acid, is passed through the tube. The hydrogen gas, before it is passed through the reduction tube, is dried by passing it through strong sulphuric acid and then through "a tube eighteen inches long" filled with fragments of calcium chloride. "The farther end of the

iron tube is connected by a cork with a bent tube dipping under water; and when the hydrogen is observed to pass through the water at about the rate that it bubbles through the sulphuric acid, the furnace is to be allowed to cool down to the temperature of the atmosphere, a slow current of hydrogen being still continued."

Reduced iron must be kept in small, dry, tightly-stoppered bottles.

Reaction. $Fe_2O_3+3H_2=2Fe+3H_2O$.

Notes. The thoroughly dried ferric hydroxide (it is rendered partly basic in drying, and soon becomes entirely converted into ferric oxide when heated) must be in fine powder. This is placed in an iron tube (such as a gun barrel, or a tube constructed expressly for this operation). Hydrogen is then passed through the tube over the basic ferric hydroxide. The hydrogen must be pure and is therefore passed through solution of lead acetate to remove sulphur, through copper sulphate or silver nitrate solution to remove arsenic and phosphorus, and through concentrated sulphuric acid to remove moisture, before it enters the iron tube. When the gas has passed over the ferric hydroxide for a minute so that the tube is filled with the gas, heat is applied at the point where the ferric hydroxide is placed, and the temperature is gradually raised. At about 350° C. the ferric hydroxide is reduced to ferroso-ferric oxide; at about 500° C. to ferrous oxide; and at about 700° C. to metallic iron. It is necessary, therefore, that the temperature should be sufficiently high, for if the product should contain much ferrous oxide it will become self-igniting in contact with air. On the other hand, if heated too strongly, the iron particles cake together and incomplete reduction will be the result. The tube and its contents should, therefore, be heated to "dull red heat"—not to a "bright red heat."

The heating must be continued until the gas after passing through the tube no longer deposits moisture on a glass plate or other suitable cold object held near the end of the reduction tube. The reduction is then completed and the heat is to be withdrawn; but the current of hydrogen through the tube must be continued until the tube and contents have cooled off, for if the reduced iron, while still hot, comes in contact with air, it might ignite and become oxidized again.

Description.—A very fine, grayish-black, lustreless, odorless and tasteless powder. The Americal Pharmacopœia requires it to contain at least 80 per cent of metallic iron; the British Pharmacopœia 75 per cent.

IRON (FERRIC) ACETATE SOLUTION.

LIQUOR FERRI ACETATIS.

An aqueous solution of ferric acetate ($Fe(C_2H_3O_2)_3=233$) containing about 31 per cent of the anhydrous salt, corresponding to about 7.5 per cent of iron.

Solution of ferric sulphate.....	100 parts
Glacial acetic acid	26 parts
Ammonia water	85 parts
Distilled water, sufficient.	

To the ammonia water diluted with 300 parts of cold water add, constantly stirring, the solution of ferric sulphate previously diluted with 1000 parts of cold water. Pour the whole on a wet muslin strainer, allow the precipitate to drain, then return it to the vessel and mix it intimately with 600 parts of cold water; again drain it on the strainer, and repeat the washing with successive portions of cold water until the washings are no longer affected by sodium cobaltic nitrate test-solution (showing the removal of ammonia and its salts). Transfer the mixture to a wet muslin strainer, allow the precipitate to drain completely, and press it, folded in the strainer, until its weight is reduced to 70 parts or less by expressing the water from it. Now add the pressed ferric hydroxide gradually to the glacial acetic acid in a tared jar provided with a glass stopper, stirring the mixture after each addition until each portion added has been nearly dissolved before another portion is added. Finally, when all of the ferric hydroxide has been added, and no more of it dissolves, add enough distilled water to make the product weigh 100 parts, mix well, allow the mixture to become clear by subsidence, and decant the clear solution, or draw it off by means of a syphon.

Solution of acetate of iron should be kept in well stoppered bottles, protected from light, in a cool place.

Reaction. First, $Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4$;

then, $Fe(OH)_3+3HC_2H_3O_2=Fe(C_2H_3O_2)_3+3H_2O$.

Notes. In order to make this preparation successfully, the directions must be strictly obeyed. Cold liquids must be used throughout, and if practicable the solution should be made in cool weather. See the notes on ferric hydroxide.

The washings may be tested with either barium chloride or sodium cobaltic nitrate. When the hydrate has been well washed and drained, as much as possible of the water held by the magma must be forcibly pressed out, and the weight of the hydroxide thus reduced to the quantity stated. This is necessary because a larger quantity of moisture retained in the magma would so dilute the acetic acid that it could no longer dissolve the hydroxide, so that a turbid mixture instead of a clear solution would result. No heat must be employed, because heat decomposes the ferric hydroxide, rendering it insoluble, whilst it at the same time volatilizes acetic acid. Even the elevation of temperature caused by the solution of the ferric hydroxide in the strong acetic acid is too great not to require to be kept under control so far as practicable. To obviate this generation of heat, the ferric hydroxide should be added in small portions at a time to the acid, as directed.

Filtration is impracticable, or perhaps worse than useless. If the solution is unclear, it will be rendered more so by any exposure to the air during filtration, from loss of acetic acid, and chemical changes, and even if the filtration be carried out so carefully as to effectually prevent loss of acid and access of air, the liquid will pass through the filter paper extremely slowly and rarely clearer than before.

The preparation is used in making tincture of acetate of iron.

Several pharmacopœias prescribe ferric chloride instead of ferric sulphate, to produce the ferric hydroxide with which the acetic acid is to be saturated.

The solution of ferric acetate of the Pharmacopœia of the United States would seem to be unnecessarily strong. A solution

representing 5 per cent of its weight of metallic iron is more easily obtained (see German formula below).

Description.—The solution of acetate of iron of the American Pharmacopœia is a dark brown-red, clear liquid of acetous odor, sweetish-acidulous, mildly styptic taste, and slightly acid reaction. Sp. w. about 1.160 at 15°.

This preparation is about 50 per cent stronger than that of the German and Swiss pharmacopœias, both of which contain a solution corresponding in strength to from 4.8 to 5 per cent of metallic iron, while the American preparation contains about 7.5 per cent of iron.

The "Solution of Ferric Acetate" of the British Pharmacopœia contains only 1.65 per cent of metallic iron. Thus 1000 parts by weight of the American solution equals 4545 parts of the British solution of the same name.

German Official Formula (Ed. III).

[The Swiss preparation is the same.]

Solution of ferric chloride, U.S.P.....	20 parts
Ammonia water	27 parts
Acetic acid, U.S.P.....	17 parts
Distilled water, sufficient.	

Dilute the solution of ferric chloride with 125 parts of distilled water, and the ammonia water with 500 parts. Add the iron solution slowly and during constant stirring to the dilute ammonia solution. Wash the precipitate in the usual manner with cold distilled water. Let the precipitate drain and express as much of the water from it as possible by means of strong pressure.

Put the moist mass of ferric hydroxide thus obtained into a suitable vessel with the acetic acid, and set the mixture aside in a cool place, shaking it frequently. When solution has been effected, or but a slight residue remains undissolved, filter the liquid and add enough distilled water to make the specific weight of the product from 1.087 to 1.091.

This solution represents from 4.8 to 5 per cent of Fe. It is, therefore, about two-thirds the strength of the solution of ferric acetate of the U.S.P.

Iron Acetate Tincture.

TINCTURA FERRI ACETATIS (U.S.P., 1880).

Solution of ferric acetate	86 ml
Acetic ether	45 ml
Alcohol	74 ml

Mix the alcohol and acetic ether; then add the solution of ferric acetate slowly and in small quantities at a time.

Notes. If the ferric acetate is added all at once, heat is generated, whereby acetic acid and acetic ether are volatilized, and the preparation is liable to become unclear from basic ferric acetate.

Must be kept in a well stoppered bottle in a cool, dark place.

The "Tinctura Ferri Acetici Aetherea" of the German Pharmacopœia is a mixture of 8 parts of the solution of acetate of iron (G.P), 1 part of alcohol (86%), and 1 part of acetic ether. It is nearly identical with the American tincture (1880) as to the percentage of iron [the American tincture corresponding to about 3.75% and the German to about 3.90% of metallic iron].

The tincture of ferric acetate was dropped from the American Pharmacopœia in the revision of 1890; but the solution of ferric acetate was retained.

Basham's Mixture.

[SOLUTION OF IRON AND AMMONIUM ACETATE.]

Tincture of ferric chloride	20 ml
Diluted acetic acid	30 ml
Solution of ammonium acetate	200 ml
Aromatic elixir	100 ml
Glycerin	120 ml
Water, sufficient.	

Pour the solution of ammonium acetate into a suitable container and add to it, successively, the diluted acetic acid, tincture of ferric chloride, elixir, and glycerin, and, finally, enough water to make the total product measure 1 liter.

This preparation should be prepared only as wanted.

Notes. The solution of ammonium acetate used should not be alkaline. The quantity prescribed of diluted acetic acid is sufficient to prevent any turbidity. As the tincture of ferric chloride is added the color changes to a decidedly red because of the formation of ferric acetate. The preparation should be perfectly clear.

IRON ALBUMINATE.

[Ferrated Albumin.]

Solution of ferric oxychloride (containing 3.5% of Fe)	300 parts
Dried egg albumen	75 parts
Solution of sodium hydroxide (containing about 0.1% of NaOH).	
Distilled water, of each sufficient.	

Dilute the solution of ferric oxychloride with 10,000 parts of water of the temperature of 50° C.

Dissolve the egg albumen in 10,000 parts of distilled water of the same temperature as before.

Pour the solution of egg albumen slowly and with constant stirring into the solution of iron.

Neutralize the mixture carefully and exactly with solution of sodium hydroxide.

The iron albuminate, which will now be completely precipitated, is to be washed with distilled water until the washings give no further reaction for chloride (with $AgNO_3$). Collect the precipitate on a cloth strainer, let it drain well, squeeze out as much of the water contained in it as can be pressed out of it by gentle pressure. Spread it on glass plates, and dry at from 40° to 50° C.

Description.—Red-brown scales or powder, odorless, of feebly ferruginous taste, forming a clear solution with water containing about 0.15% of sodium hydroxide in solution. It contains about 20% of Fe.

Solution of Albuminated Iron.

[After the Danish Pharmacopœia.]

Dried egg albumen	10 parts
Ferric chloride	60 parts
Cinnamon water	100 parts
Alcohol	50 parts
Distilled water, sufficient.	

Dissolve the egg albumen in 350 parts of distilled water at a temperature of about 40° C. Strain the solution.

Dissolve the ferric chloride in 430 parts of distilled water.

Pour the solution of egg albumen slowly, and during uninterrupted stirring, into the solution of ferric chloride.

Heat the mixture over a water-bath at a temperature of 90° C. for half an hour.

Then let it cool and add to it the cinnamon water and alcohol, previously mixed. Finally add enough distilled water to make the total weight of the final product 1000 parts.

Description.—A red-brown liquid, which appears clear by transmitted light, but unclear by reflected light. It has scarcely any chalybeate taste.

Reduced to one-half its volume by evaporation it exhibits an acid reaction.

When heated to the boiling point it is not rendered turbid, nor does the addition of alcohol make it unclear (absence of albumin).

If a few drops of ammonia water or diluted hydrochloric acid be added to 5 Gm of the solution of ferrated albumin a red-brown precipitate is formed. But 5 Gm of the solution shaken with 15 Gm of ammonia water produces a clear mixture.

Potassium carbonate causes it to gelatinize.

The preparation contains about 0.4 per cent of Fe.

Alkaline Solution of Iron Albuminate.

[After the Swiss Pharmacopœia.]

Fresh egg albumen	200 parts
Solution of oxychloride of iron (3.5% Fe)	120 parts
Solution of sodium hydroxide (5% NaOH)	15 parts
Alcohol	150 parts
Cinnamon water	100 parts
Distilled water, sufficient.	

Mix the egg albumen with 4000 parts of distilled water heated to a temperature of 50° C.

Mix the solution of ferric oxychloride with 4000 parts of distilled water heated to a temperature of 50° C.

Pour the solution of egg albumen slowly and with uninterrupted stirring into the diluted solution of oxychloride of iron.

Add carefully enough solution of sodium hydroxide (of 0.15% strength) to render the liquid perfectly neutral.

Wash the precipitated ferrated albumin with distilled water having the temperature of 50° C. until the washings no longer give any reaction for chloride.

Collect the precipitate on a strainer and let it drain until its weight is reduced to 400 parts.

Transfer the ferrated albumin to a bottle, add the solution of sodium hydroxide to it, and mix well.

Then add the alcohol and cinnamon water previously mixed, and finally enough distilled water to make the total weight of the product 1000 parts.

Let it stand until any solid particles shall have subsided, and then decant the clear liquid, or filter the product if necessary.

IRON ARSENATE.

FERRI ARSENAS.

Ferrous sulphate	46 parts
Sodium arsenate, dried at 149° C.	35 parts
Sodium bicarbonate	10 parts

Dissolve the sodium arsenate in 200 parts of boiling distilled water, and the ferrous sulphate in 240 parts of boiling water; add the solution of the iron salt to that of the arsenate; then add the sodium bicarbonate previously dissolved in 120 parts of hot water. Stir well. Collect the precipitate on a wetted muslin strainer and wash it with water until the washings cease to be affected by test solution of barium chloride. Press out the water from the washed precipitate, folded in strong linen, in a screw press, and dry the product on porous bricks in a warm air chamber at a temperature not exceeding 40° C.

Notes. The reaction is analogous to that occurring in the process of preparing the precipitated ferroso-ferric phosphate. The precipitate is ferroso-ferric arsenate, of variable composition and color.

Description.—A green to greenish-blue, odorless and tasteless powder, insoluble in water and alcohol.

IRON BENZOATE.

FERRI BENZOAS.

To a solution of ferric chloride add ammonia water gradually as long as no precipitate is formed. Then add a solution of ammonium benzoate until precipitation is completed. Collect the precipitate, wash it with a limited amount of cold water, let it drain somewhat, press out the remaining water as far as practicable, and then dry the product without the aid of heat.

Another Method.

Sodium benzoate	5 parts
Ferric chloride	3 parts
Distilled water, sufficient.	

Dissolve the sodium benzoate in 25 parts of distilled water, and the ferric chloride in 40 parts of distilled water.

Pour the solution of ferric chloride slowly into the solution of sodium benzoate, stirring constantly.

Wash the precipitate well with distilled water, let it drain, and dry it.

Description.—The product is a (brownish) flesh-colored, odorless and tasteless powder. Sparingly soluble in fixed oils.

IRON BROMIDE.

FERRI BROMIDUM.

$FeBr_2$=216.

Ferrous bromide in water solution is obtained when iron and bromine react upon each other in water, the iron being used in excess. The solution is bright green. Ferrous bromide in the solid state can not be kept, nor does a water-solution keep without sugar.

Syrup of Bromide of Iron.

A *syrup of ferrous bromide* is sometimes used, which contains 10 per cent of the bromide, keeps well, and may be prepared as follows:

Iron, in the form of wire, cut into small pieces.	30 Gm
Bromine	75 Gm
Sugar, in coarse powder	600 Gm
Distilled water, sufficient.	

Introduce the iron into a flask, add 200 ml of distilled water, and afterward the bromine. Shake the mixture occasionally, until the reaction ceases and the solution has acquired a green color and has lost the odor of bromine. Place the sugar in a porcelain dish and filter the solution of bromide of iron into the sugar. Rinse the flask and iron wire with 90 ml of distilled water, and pass the washings through the filter into the sugar. Stir the mixture with a glass rod, heat it to the boiling point on a sand-bath, and, having filtered the syrup through paper into a tared bottle, add enough distilled water to make the product weigh 1000 Gm. Lastly, shake the bottle, which should be completely filled, securely stoppered, and kept in a place accessible to daylight.

Notes. The bromine must be added cautiously, as the reaction is sometimes violent. When too violent, the chemical action can be controlled by keeping the flask in cold water.

IRON SACCHARATED CARBONATE.

FERRI CARBONAS SACCHARATUS.

Ferrous sulphate	50 parts
Sodium bicarbonate	35 parts
Sugar, in fine powder, sufficient.	

Dissolve the ferrous sulphate in 200 parts of boiling distilled water, and the sodium bicarbonate in 500 parts of distilled water of the temperature of about 40° to 50° C. Filter the solutions separately. Put the bicarbonate solution into a flask capable of holding 1000 parts of water; then add the hot solution of ferrous sulphate, agitating the mixture well, and when the effervescence has subsided fill the flask at once with boiling distilled water, cork it to exclude air, and set it aside for two hours. Then remove the mother liquor by means of a siphon, and again fill the flask with boiling distilled water, shake it well, let it settle once more, and repeat the washing in the same manner until the wash water produces but little turbidity with test solution of barium

chloride. Then transfer the precipitate to a wetted muslin strainer, let it drain, or press out the water, and mix the magma intimately with 80 parts of sugar in a porcelain dish, evaporate the mixture to dryness by means of a water-bath, powder the product, adding more dry powdered sugar if necessary to make the total weight of the product 100 parts, and keep it in a tightly corked bottle.

Reaction. $FeSO_4+2NaHCO_3=FeCO_3.H_2O+Na_2SO_4+CO_2$.

Notes. All the precautions directed by this formula are intended to prevent the oxidation of the precipitate by contact with the air. Sodium bicarbonate is substituted for the normal carbonate in order that the escaping carbonic acid may help to exclude the air. If about 5 parts of sugar be added to the solution of bicarbonate of sodium before the iron salt is poured into it, and if the wash water also be sweetened with about four per cent of sugar, the oxidation is still more effectually prevented. Boiling water is used in order that no air may be contained in it; and the whole process must be expeditiously carried to its completion in order not to expose the product to oxidation longer than necessary. When the precipitate has been mixed with the sugar, the mixture should not be stirred any more than is necessary during the process of its evaporation to dryness, and as soon as the mass is sufficiently dry it should be at once powdered and put into small bottles, which are to be immediately and tightly closed.

Hager recommends the use of milk sugar instead of cane sugar to facilitate the drying. The German Pharmacopœia prescribes one-fourth of milk sugar and three-fourths of cane sugar instead of cane sugar alone.

A more dense precipitate is obtained by having the solutions hot when mixed. But the solution of sodium bicarbonate can not be made with water of a higher temperature than 50° C., because the bicarbonate decomposes if subjected to a much higher temperature than that.

Sugar and honey aid in preventing oxidation, by excluding the air from the product.

See also the notes under the title, Mass of Carbonate of Iron.

Description.—A grayish or greenish-brown powder, which oxidizes on exposure to the air.

Mass of Carbonate of Iron.

[PILL OF CARBONATE OF IRON. VALLET'S MASS.]

Ferrous sulphate	100 parts
Sodium carbonate	110 parts
Clarified honey	38 parts
Sugar, in coarse powder	25 parts
Syrup, distilled water, each sufficient.	

Dissolve the sulphate of iron and the carbonate of sodium separately, each in two hundred parts of boiling distilled water, and, having added twenty-five parts of syrup to the solution of the iron salt, filter both solutions. Mix when cold, in a bottle just large enough to hold the mixture, or add enough distilled water to fill it; cork the bottle well, and set it aside, that the carbonate of iron may settle. Pour off the supernatant liquid, and, having mixed syrup and distilled water in the proportion of one part of syrup to sixteen parts of water, wash the precipitate with the mixture until the washings no longer have a saline taste. Drain the precipitate on a flannel cloth, and express as much of the water as possible. Lastly, mix the precipitate immediately with the honey and sugar, and, by means of a water-bath, evaporate the mixture, constantly stirring, until reduced to one hundred parts.

Notes. The reaction is given under the title, Subcarbonate of Iron. Compare this process with that for saccharated carbonate of iron. The ferrous sulphate must be in crystals in no degree effloresced. Sodium bicarbonate may be used here as well as in the preceding preparation instead of the normal carbonate; 75 parts of bicarbonate should be substituted for the 110 parts of carbonate. If bicarbonate is used the solution of it must be made not with boiling water but with water of a temperature not exceeding 50° C. The use of hot solutions renders the precipitate more dense, which is of material advantage in the preparation of saccharated ferrous carbonate and Vallet's mass, where expeditious washing is necessary.

Description.—A soft, dark grayish-brown mass, having a sweet, afterwards ferruginous, taste.

IRON (FERRIC) CHLORIDE.

FERRI CHLORIDUM (RUBRUM).

$$FeCl_3+6H_2O=270.2.$$

Iron, in the form of fine, bright wire, and
cut into small pieces ..... 15 parts
Hydrochloric acid,
Nitric acid,
Distilled water, each, a sufficient quantity.

Put the iron into a flask capable of holding two or three hundred parts of water. Add 54 parts of hydrochloric acid diluted with 25 parts of distilled water.

Let the mixture stand in a warm place until effervescence has nearly ceased. Then heat it to the boiling point and continue the boiling for two or three minutes.

Let the liquid cool somewhat and then filter it, while still hot, through paper; and, having rinsed the flask and undissolved iron with a little hot distilled water, pass the rinsings also through the filter.

Add 28 parts of hydrochloric acid to the filtrate; add this mixture, a little at a time, to 8 parts of nitric acid contained in a capacious porcelain dish, gently warmed, waiting after each addition until the copious evolution of red nitrous vapors subsides before adding more. When all of the iron solution has been added to the nitric acid, and active effervescence has ceased, heat the dish and contents by means of a sand-bath until the liquid is free from nitrous odor.

Then test a few drops of the liquid, diluted with water, with freshly prepared potassium-ferricyanide test-solution. Should this reagent produce a blue precipitate or a blue color, add a little more nitric acid, drop by drop, to the hot solution of ferric chloride in the porcelain dish, as long as any further evolution of red vapors is observed. The solution is then tested as before, and when the test-solution of potassium ferricyanide no longer produces a decidedly blue color, showing that only a trace of ferrous chloride remains in the liquid, no more nitric acid must be added.

[Should an excess of nitric acid be found to have been added, the excess must be expelled by heating the liquid until all odor of nitric acid has ceased.]

Then add 5 parts of hydrochloric acid and enough distilled water to make the whole weigh 60 parts.

Set the dish aside, covered with glass, in a cool place, until a solid crystalline mass is formed of the contents.

Remove the crystalline mass, break it into pieces, and keep the product in glass-stoppered bottles, protected from light.

Reactions. First, $2Fe+4HCl=2FeCl_2+2H_2$; then, $6FeCl_2+6HCl+2HNO_3=3Fe_2Cl_6+4H_2O+2NO$.

Notes. The iron is readily dissolved by the hydrochloric acid, especially in the beginning; as the solution becomes charged with ferrous chloride, however, the action is slower and it is advantageous to promote it by the aid of heat. In order that the density of the solution of ferrous chloride may not be so great as to retard the chemical action, the Pharmacopœia directs that the hydrochloric acid be diluted before the iron is added. The evolution of hydrogen may cease before all the HCl is decomposed by the iron at the ordinary temperature, but when the liquid is brought to the boiling point the acid is completely saturated.

As the amount of hydrochloric acid saturated with iron to form ferrous chloride determines the strength of the finished solution and the amounts of hydrochloric and nitric acids required to raise that ferrous chloride to ferric chloride, it is important that the whole quantity of hydrochloric acid first used be completely saturated.

Some ferric chloride is formed before the acid is saturated, especially if the iron was rusty; but after boiling the liquid with the excess of iron present the solution contains ferrous salt only.

The solution of ferrous chloride is then to be treated with hydrochloric and nitric acids to convert it into ferric salt. When the solution is concentrated this may be accomplished at little above ordinary room temperature. When the solution is dilute, on the other hand, a temperature near the boiling point is required to complete the reaction. A good rule is to evaporate the solution of ferrous chloride until it has about 1.30 sp. w., and after adding the additional quantity of hydrochloric acid prescribed, to add this solution slowly, a little at a time, to the nitric acid. If all of the solution of ferrous chloride is added at once, the reaction is too violent, the red fumes being evolved so copiously and suddenly

that the liquid may boil over. The most suitable temperature at which the reaction may be carried out is between 80° and 90° C.

A small excess of nitric acid is unavoidable if the solution is to be entirely free from ferrous salt. A greenish-brown coloration with test-solution of potassium ferricyanide is allowable, indicating merely traces of ferrous salt. But the excess of nitric acid even then liable to be present requires considerable evaporation to get rid of it. Long continued strong heating of the solution of ferric chloride (to drive off HNO_3) is liable to result in decomposition with the formation of basic ferric chloride. When the solution of ferric chloride is perfectly free from nitrous odor, and contains not more than traces of ferrous salt, the last addition of hydrochloric acid is made, and the distilled water added. This hydrochloric acid is added to prevent the formation of basic chloride.

The Pharmacopœia directs that the solution shall be evaporated down to given quantity by weight. That quantity is the amount of crystallized ferric chloride produced by the ferrous chloride formed by the saturation of the hydrochloric acid first used (54 parts) with the iron.

It may preferably be evaporated down until a small sample taken out and cooled no longer gives off acid vapors in the air, nor has a pronounced odor of HCl, and the liquid has about 1.62 to 1.65 sp. w. at 25° C. This procedure is always necessary in cases where any portion of the liquid has been lost by accident at any stage of the process.

The chloride crystallizes readily, even within twelve hours, if of proper strength; but may not solidify for several weeks if too strong. When the evaporation has been carried too far, the liquid does not contain enough water of crystallization, and in that case it must be placed in a cool, moist atmosphere, from which the necessary additional moisture may be absorbed. A solution containing just enough hydrochloric acid crystallizes more readily than one that is not acid enough, and yields a lighter-colored and harder crystalline mass. The crystallization also proceeds more satisfactorily in a loosely covered dish than in a closely covered vessel.

Should the crystalline mass of ferric chloride stick to the dish so tenaciously that its removal may involve the danger of breaking

the vessel, this difficulty may be avoided by gentle warming, whereby the cake is loosened.

The product ought to be dried over sulphuric acid or over quick-lime.

The ferric chloride is used chiefly for preparing aqueous and alcoholic solutions.

Description.—Orange yellow, crystalline masses or pieces, having a faint odor of hydrochloric acid (sometimes odorless when quite dry), and a strongly styptic taste. Deliquescent in moist air. Freely soluble in water and in alcohol.

Ferrated Ammonium Chloride.

[FLORES MARTIS.]

Ferric chloride	2 parts
Ammonium chloride	18 parts
Diluted hydrochloric acid	1 part
Distilled water	60 parts

Dissolve, filter, and evaporate the filtrate over a water-bath, during constant stirring, to dryness.

Put the dry product at once in a glass stoppered bottle and keep it protected from light.

An orange yellow, deliquescent powder, which is not a chemical compound but a mechanical mixture of the two chlorides.

It contains about 2 per cent of Fe.

Notes. The Swiss Pharmacopœia orders 77 parts of solution of ferric chloride (U. S. P.) to 600 parts of ammonium chloride, with 330 parts of water, and without any hydrochloric acid; this solution to be evaporated to dryness.

The German Pharmacopœia orders about 7 parts of solution of ferric chloride (U. S. P.) and 32 parts of H_4NCl.

Iron (Ferric) Chloride Solution.

LIQUOR FERRI CHLORIDI; U. S.

An aqueous solution of ferric chloride, containing 37.8 per cent of the anhydrous salt [$FeCl_3$=162.2] corresponding to 62.9 per cent of the crystallized chloride [$FeCl_2+6H_2O$=270.2], or to about 13 per cent of metallic iron, and containing also some

free hydrochloric acid [about 1.6 per cent of HCl, equivalent to 5 per cent of the official hydrochloric acid].

The official solution of ferric chloride is prepared from iron, hydrochloric acid, nitric acid, and water as described under the title of Iron Chloride. The quantities of the materials required to make one hundred parts of the finished solution are:

Iron, in the form of fine bright wire, cut into small pieces..... 15 parts
Hydrochloric acid..... 87 parts
Nitric acid.
Distilled water, each, sufficient.

The iron is put into a flask capable of holding at least twice the volume of the finished product to be made; 54 parts of hydrochloric acid and 25 parts of distilled water are added; the complete saturation of the acid is insured by the aid of heat, and the ferrous chloride is converted into ferric chloride by means of 28 parts of hydrochloric acid and 8 parts of nitric acid, precisely as described under the title of ferric chloride. The solution is tested, and when it no longer contains any ferrous salt (or when it contains only traces of ferrous chloride), and the excess of nitric acid has been dissipated by heat so that no nitrous odor is retained by the liquid, the last 5 parts of hydrochloric acid is added together with enough distilled water to make the whole weigh 100 parts (instead of only 60 parts as prescribed in the formula for making solid crystalline ferric chloride).

Notes. The reactions, and the several steps of the process, are explained under the title, Ferric Chloride.

A solution of ferric chloride cannot be permanent without the presence of free hydrochloric acid. In the absence of a sufficient quantity of HCl, a basic ferric chloride is formed, which precipitates. To redissolve this yellow basic salt requires more hydrochloric acid than it takes to prevent its formation. Continued heating results in the loss of HCl and the formation of yellow oxychloride; if, however, sufficient HCl is present, no precipitate is formed, but the solution upon being heated becomes darker in color, a ferric chloride with 5 molecules of water being formed. After standing some time, however, the darkened solution resumes its original color, and then contains the chloride with 12 molecules of water. Weak solutions are more readily altered, and

when darkened by heating do not resume a lighter color on standing.

The solution of ferric chloride often contains less iron than the pharmacopœial standard of strength requires. This may be the result of the employment of hydrochloric acid not of the full official strength, or of the incomplete saturation of the acid with iron.

The preparation is frequently found to be contaminated with nitric acid, or to contain some ferrous chloride; it can not contain both ferrous salt and free nitric acid except for a very brief period during the process of preparation, and then only traces of each. The presence of a trace of ferrous chloride can not be considered as a very serious defect, for the solution is used almost exclusively for the purpose of preparing the tincture of chloride of iron which unavoidably contains a large amount of ferrous salt formed by the reducing action of the alcohol upon the ferric chloride.

But the presence of free nitric acid is more objectionable. Hence it is better to use a trifle less nitric acid than is necessary to completely remove all traces of ferrous salt, than to use a trifle too much so as to leave an excess of that acid, because the expulsion of the excess of nitric acid by heating the liquid is almost impossible without the great risk of causing the formation of basic ferric chloride by oxidation, a corresponding amount of hydrochloric acid (formed from the decomposing ferric chloride) being evolved from the hot liquid.

Sunlight slowly decomposes ferric chloride (solid as well as in solution) into ferrous chloride and chlorine. In the presence of organic substances, such as alcohol and other reducing agents, this reduction is much more rapid.

Solution of ferric chloride may also be prepared by dissolving ferric hydroxide in hydrochloric acid; but a strong solution can not be made in that way because undiluted hydrochloric acid is of only 32 per cent strength and recently precipitated ferric hydroxide contains much water which would further dilute the solution thus formed. If the official hydrochloric acid be used, and a ferric hydroxide *free from water* were employed to form the solution of ferric chloride according to the equation:

$$Fe(OH)_3+3HCl=FeCl_3+3H_2O$$

the resulting solution would still contain only about 36 per cent of $FeCl_3$.

As 1000 Gm of the official solution of ferric sulphate (containing 27.8 per cent of that salt) corresponds to 225.2 Gm of ferric chloride ($FeCl_3$) it follows that if the ferric hydroxide precipitated from 1000 Gm of the solution named is, after the washing, subjected to strong pressure and thus reduced to not over 640 Gm weight, and 486 Gm of official hydrochloric acid be then added, making the total weight of the product 1126 Gm, the solution of ferric chloride thus obtained will contain 20 per cent of $FeCl_3$, together with a very slight excess of HCl (less than 0.2%).

When the solution of ferric chloride is prepared from a solution of ferrous chloride, the conversion of the ferrous chloride into ferric chloride may be effected by conducting chlorine gas into the solution of ferrous chloride. This method is advantageous because it affords a product absolutely free from both $FeCl_2$ and HNO_3.

Some pharmacopœias direct that the solution of ferric chloride be made by dissolving the crystalline ferric chloride in distilled water, which is a direct, convenient and definite method.

The *official solution of ferric chloride (U. S. P.)*, containing slightly more than 13 per cent of Fe, corresponding to 37.8 per cent of $FeCl_3$, or 62.9 per cent of $FeCl_3.6H_2O$, or to 18.63 per cent Fe_2O_3, or to 24.93 per cent of ferric hydroxide, compares in iron strength to the official solution of ferric sulphate (U. S. P.) as 162 to 100; in other words 100 parts of solution of ferric chloride contains as much iron as is contained in 162 parts of solution of ferric sulphate. In preparing ferric hydroxide from solution of ferric chloride, 100 parts of the solution will require about 135 parts of official ammonia water (10%) to insure a sufficient excess of ammonia.

The *solution of ferric chloride of the German and Swiss Pharmacopoeias* contains 10 per cent of Fe; hence about 77 parts of that solution equals 100 parts of the solution of the U. S. P.

The Danish and Norwegian Pharmacopoeias contain a solution made of equal parts of *crystallized* ferric chloride and distilled water.

Thus that solution is nearly identical with that of the German and Swiss Pharmacopœias.

The "liquor ferri perchloridi fortis" of the *British Pharma-*

copoeia (1898) has the sp. w. 1.42 and 100 ml of it contains 22.5 grams of iron; in other words, 142 grams of the solution contains 22.5 grams of iron, which corresponds to 15.845 per cent of metallic iron. Thus 820 grams of the British "strong solution of ferric chloride" exactly equals 1000 grams of the American "solution of ferric chloride." But the British Pharmacopœia also contains a weaker preparation called "solution of ferric chloride" (the English title being identical with that of the American preparation), which is made by mixing 1 volume of the strong solution of ferric chloride with 3 volumes of distilled water and, therefore, contains an amount of ferric chloride corresponding to 5.09 per cent of metallic iron. Thus 2552 grams of the British solution of ferric chloride equals 1,000 grams of the American solution of ferric chloride.

The British "*strong solution of ferric chloride*" contains about 46 per cent of anhydrous ferric chloride, corresponding to 76.54 per cent of crystallized ferric chloride. The British "solution of ferric chloride" contains about 14.83 per cent of anhydrous ferric chloride, corresponding to about 24.68 per cent of the crystallized salt ($FeCl_3.6H_2O$).

The solution of ferric chloride of the *Pharmacopoeia of the Netherlands* is prepared by dissolving 3 parts of ferric chloride in 1 part of water. It is described as containing 14.4 to 15.5 per cent of Fe, and having the sp. w. 1.441 to 1.488.

Description.—The solution of ferric chloride of the American Pharmacopœia is a reddish-brown liquid having but a faint odor (and that the odor of hydrochloric acid only). It has a strongly styptic ferruginous taste and an acid reaction. Sp. w. 1.387 at 15°.

Method after the Swiss Pharmacopoeia (1893).

Iron, in fine, bright wire, cut in small pieces	1 part
Hydrochloric acid (32% of HCl)	4 parts
Distilled water, sufficient.	

Put the iron and hydrochloric acid in a suitable vessel of glass or porcelain in a warm place. When effervescence has ceased heat the liquid at the boiling point for a few minutes. Filter the solution.

Conduct a current of washed chlorine into the solution until the liquid no longer produces a blue precipitate with test-solution of potassium-ferricyanide.

Then evaporate the solution until reduced to five times the weight of the iron dissolved, cover it and set it aside in a cool place to crystallize. Dry the crystalline mass over sulphuric acid (or over lime), and dissolve the ferric chloride, thus dried, in an equal weight of distilled water.

This solution contains about 50 per cent of crystallized ferric chloride corresponding to about 10 per cent of metallic iron.

Notes. To ascertain the amount of iron dissolved in the hydrochloric acid, the undissolved portion of the metal must be collected and weighed and its weight deducted from the quantity originally put into the acid.

The solution of ferrous chloride may, if most convenient, be put into a series of Woulff bottles connected with each other and with the wash-bottle through which the chlorine gas is passed from the generator. Only the solution in the last Woulff bottle need be tested with the solution of potassium-ferricyanide.

It is well, also, to connect the last Woulff bottle with another bottle containing sodium carbonate solution in order to fix the excess of chlorine passing beyond the last Woulff bottle so as to prevent its escape into the room.

The Pharmacopœia of the Netherlands, also, directs the use of chlorine to "oxidize" the ferrous chloride to ferric.

It will be seen that by this process we first make crystallized ferric chloride, and then make the solution out of that finished chloride. It would, therefore, be as well to divide the working formula accordingly into two—giving one formula for the preparation of the dried crystallized ferric chloride, and a separate one for the preparation of the solution.

Iron (Ferric) Chloride Tincture.

TINCTURA FERRI CHLORIDI.

The official tincture of chloride of iron is "a hydroalcoholic solution of ferric chloride [$FeCl_3$=162.2], containing about 13.6 per cent of the anhydrous salt," corresponding to 22.6 per cent

of crystalline ferric chloride ($FeCl_3+6H_2O=270.2$), or to about 4.7 per cent of metallic iron.

[It should be understood, however, that the official preparation contains not only ferric chloride, alcohol and water, but also a large proportion of ferrous compound together with aldehyde and chlorinated ethereal compounds formed by reaction between the alcohol and the ferric chloride.]

It is prepared as follows:

Solution of ferric chloride..... 250 volumes
Alcohol, sufficient to make the product 1000 volumes.

Mix the liquids, and let the mixture "stand, in a closely covered vessel, at least three months; then transfer it to glass-stoppered bottles, and keep it protected from light."

Notes. We are informed that the object of letting the mixture of alcohol and solution of ferric chloride stand for "at least three months" is the formation of "certain chlorinated ether compounds."

Several writers attribute the formation of the ethereal compounds to the reaction of the free hydrochloric acid (contained in the solution of the ferric chloride) upon the alcohol; but this can not be true because a liquid containing only 1.6 per cent of HCl does not react upon alcohol at all.

The ethereal products are formed by reaction between the ferric chloride and the alcohol, and the principal reactions are probably the following:

$$FeCl_3+3C_2H_5OH=Fe(OH)_3+3C_2H_5Cl, \text{ and}$$

$$2FeCl_3+C_2H_5OH=2FeCl_2+2HCl+C_2H_4O.$$

The ferric hydroxide formed by the reaction first stated is not at once precipitated. It remains dissolved in the liquid forming a soluble compound with the undecomposed portion of the ferric chloride. But a "basic ferric chloride" or "oxychloride" of iron is finally thrown down.

That aldehyde and ethyl chloride are both contained in a tincture of ferric chloride may be at once discovered even by the sense of smell.

The hydrochloric acid formed simultaneously with the aldehyde is not sufficient to form $FeCl_3$ with all the $Fe(OH)_3$ formed with the ethyl chloride.

As the quantity of $FeCl_3$ diminishes and the amount of $FeCl_2$ increases, the preparation becomes materially lighter in color.

These reactions take place very slowly or not at all when the mixture is kept in a dark place; they progress rapidly when it is exposed to direct sunlight.

The Pharmacopœia says that "after the tincture has been exposed for some time to daylight it yields a greenish or greenish-blue color with potassium-ferricyanide test-solution, showing the presence of some ferrous salt due to reduction." But while the formation of the ethyl chloride goes on most rapidly when the tincture is freely exposed to light, it can not proceed far without resulting in the separation of iron (as basic ferric hydroxide and oxychloride), and consequently in the diminution of the iron strength of the preparation.

The Pharmacopœia fails to state, but probably intends, that the mixture of alcohol and solution of ferric chloride should be protected against light during the prescribed three months standing as well as afterwards. If thus protected the preparation will contain very little, if any, of either ethyl chloride or aldehyde. If, on the other hand, the presence of these ethereal substances is desirable, exposure to light would seem to be necessary to obtain any considerable quantity of them.

When tincture of ferric chloride is exposed to full direct sunlight for a sufficiently long time it finally acquires a very pale amber color, or becomes almost colorless. Preparations so made, sometimes containing added ether and sometimes not, were and are to be found in the pharmacopœias of various countries.

The addition of the citrate of either potassium, sodium or ammonium, changes the color of the tincture of ferric chloride to a bright green and also removes the styptic taste. A preparation called "tincture of citro-chloride of iron" [and, sometimes, very incorrectly, "taste-less tincture of chloride of iron"] is contained in the National Formulary of the American Pharmaceutical Association. What iron-compound this preparation contains is not known.

Description.—The American tincture of chloride of iron is a clear brownish-red liquid having a peculiar odor suggestive of

aldehyde and ethereal compounds. It has a styptic ferruginous taste and an acid reaction. Sp. w. 0.960 at 15°.

Tinctura Ferri Chlorati Aetherea

of the German Pharmacopœia is prepared as follows:

Solution of ferric chloride (G. P.)	1 part
Ether	1 part
Alcohol (86%)	7 parts

Mix the liquids, put the mixture in a bottle of white glass and set it in full sunlight until wholly decolorized. Then put the bottle in a shaded place, and remove the stopper occasionally to admit air, until the liquid acquires a yellow color.

Notes. Light causes reaction between the ferric chloride and alcohol, resulting in the formation of ferrous chloride, aldehyde and hydrochloric acid, and also ethyl chloride and water. When the liquid is now put in a shaded place and the stopper of the bottle removed from time to time, the ferrous chloride is oxidized by the air and some basic ferric chloride is formed which colors the solution yellow. A part of the aldehyde is at the same time oxidized to acetic acid, perhaps partly by the aid of some free chlorine formed while the liquid was exposed to light.

The finished ethereal tincture of chloride of iron, therefore, contains alcohol, ferric chloride, ferrous chloride, ethyl chloride, aldehyde, acetic acid, and basic ferric chloride.

IRON (FERROUS) CHLORIDE.

FERRI CHLORIDUM (VIRIDE).

$FeCl_2.4H_2O=198.8$.

Iron wire	1 part
Hydrochloric acid	4 parts
Distilled water	2 parts

Dissolve the iron in the acid and water, previously mixed; when effervescence has ceased, heat to boiling. Let settle, decant, wash the undissolved iron with a little water, adding the washings to the decanted liquor, filter while hot, and crystallize.

Remove the crystals from the mother liquor, drain them in a funnel, and put them at once in a bottle.

Reaction. $Fe_2+4HCl=2FeCl_2+2H_2$.

Description.—Large, bluish-green, transparent crystals. Odorless. Taste astringent, ferruginous. Readily soluble in water and in glycerin.

Oxidizes on exposure to air, and can not be effectively protected against oxidation except by submerging the crystals in some liquid in which the chloride is insoluble.

IRON (FERRIC) CITRATE.

FERRI CITRAS.

Evaporate any convenient quantity of the solution of ferric citrate over a water-bath at a temperature not exceeding 60° C. to the consistence of syrup, and spread it on glass plates to dry in scales.

Keep it in well stoppered bottles in a cool, dark place.

Description.—Thin, transparent, garnet-red scales, odorless, of slightly ferruginous and only slightly acidulous taste. Freely soluble in water; quickly in hot water. Insoluble in alcohol. It gradually loses its ready solubility in water, and more rapidly when exposed to light. The percentage of iron is from 19 to 20%, but it varies; and the percentage of moisture retained by the scales is also variable. The preparation nearly corresponds to $FeC_6H_5O_7.3H_2O$, but may contain less iron and more water than that formula indicates. It is, therefore, not a definite chemical compound.

German Official Method.

Solution of ferric chloride, U. S. P.....	20 parts
Ammonia water.....	27 parts
Citric acid.....	9 parts
Distilled water, sufficient.	

Dilute the solution of ferric chloride with 100 parts of distilled water, and the ammonia water with 75 parts of distilled water. Pour the iron solution slowly and during uninterrupted

stirring into the dilute ammonia solution. Wash the precipitate well with cold distilled water in the usual way.

Dissolve the citric acid in 35 parts of distilled water. Add the ferric hydroxide to the citric acid solution in a porcelain dish, and heat the mixture at not over 50° C., stirring frequently, until the solution is saturated or the ferric hydroxide nearly all dissolved.

Filter the solution, evaporate it at not over 50° C. to the consistence of syrup, and scale the product in the usual way (see other formulas for scale salts).

Ammonio-Ferric Citrate.

Solution of ferric citrate	3 parts
Ammonia water	1 part

Mix and evaporate the mixture over a water-bath at not over 60° C. to the consistence of thick syrup; spread this on glass plates and let it dry in scales.

Keep the product in a well stoppered bottle, in a cool, dark place.

Notes. For making this preparation some formulas prescribe that three parts of citric acid dissolved in water be saturated with ferric hydroxide as described in the notes under Solution of Ferric Citrate, and that one part of citric acid, saturated with ammonia, be afterwards added, and the solution evaporated to form scales.

In the official formula of America the proportions prescribed of the materials are: 100 volumes of solution of ferric citrate and 40 volumes of ammonia water, which proportions correspond to 3 parts and 1 part by weight.

Description.—Thin, transparent, garnet-red scales; odorless; taste slightly saline and faintly ferruginous. Completely and quickly soluble in water. Insoluble in alcohol.

Another Method.

[After the Swiss Pharmacopœia.]

Solution of ferric chloride, U. S. P.	154 parts
Citric acid	70 parts
Ammonia water.	
Distilled water, of each sufficient.	

Dilute the solution of ferric chloride with 600 parts of distilled water.

Dilute 200 parts of ammonia water with 600 parts of distilled water.

Add the iron solution gradually and with constant stirring to the dilute ammonia solution. Wash and drain the precipitate in the usual way and forcibly press out as much of the water as practicable from the magma.

Dissolve the citric acid in 140 parts of distilled water, adding enough ammonia water to produce a slightly alkaline reaction. Filter.

Dissolve the ferric hydroxide in the solution of ammonium citrate with the aid of heat applied by means of a water-bath and not exceeding 50° C.

Evaporate the solution to a syrupy consistence, spread it on glass plates, and let it dry to form scales.

Iron (Ferric) Citrate Solution.

LIQUOR FERRI CITRATIS; U. S.

Solution of normal ferric sulphate.....	105 parts
Citric acid.....	30 parts
Ammonia water (10% of H_3N).....	90 parts
Distilled water.	

Mix the ammonia water with 300 parts of cold water, and the iron solution with 1000 parts of cold water. Add the cold diluted iron solution gradually to the ammonia solution, stirring constantly. Let the precipitated ferric hydroxide subside, and decant the supernatant liquid. Pour the remaining liquid, containing the magma, upon a wetted muslin strainer; let it drain; return the precipitate to the precipitation vessel and mix it well with 1500 parts of cold water. Let settle, decant, and again drain the magma on the muslin strainer. Repeat this washing several times, until the washings give but a slight cloudiness when mixed with test-solution of barium chloride. Finally let the ferric hydroxide be well drained. Then place it in a strong press cloth and forcibly express from it, by means of a screw press, as much as possible of the water. [The magma obtained from 105

parts of solution of ferric sulphate may be pressed until the press cake weighs less than 65 parts.]

Put the citric acid in a porcelain dish and add to it about one-half its weight (15 parts) of distilled water. Place the dish over a water-bath and keep its contents at a temperature not exceeding 40°. Add the press cake of ferric hydroxide, in small portions at a time, stirring well, and allowing each portion to dissolve before adding another. When about three fourths of the ferric hydroxide has been added, raise the temperature of the contents of the dish to 50°, and when all of the ferric hydroxide has been added let the temperature be increased to 60°, and continue heating and stirring for about fifteen minutes, taking care not to allow the temperature to rise above 60°. Filter the solution and evaporate it at a temperature not exceeding 60° until it weighs 100 parts.

Keep the product in well closed bottles in a cool place and protected from light.

Reactions.

$Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4$; then

$Fe(OH)_3+H_3C_6H_5O_7=FeC_6H_5O_7+3H_2O$.

Notes. The pharmacopœial directions do not require that the water should be squeezed out of the magma of ferric hydroxide, nor that the hydroxide shall be added gradually to the citric acid mixed with a little water. The Pharmacopœia instead directs that the drained magma be at once mixed with the citric acid and the mixture heated at 60° until the precipitate (the hydroxide) is dissolved. It is better, however, to proceed as here described, adding gradually the ferric hydroxide, deprived of as much water as can be pressed out of it, and raising the temperature to 60° only at the end. By proceeding in this manner the saturation of the citric acid will be effected without exposing the ferric hydroxide or the solution of ferric citrate to that high temperature any longer than is unavoidable, and the concentration of the solution to the required standard requires but little evaporation.

Ferric hydroxide is easily decomposed and rendered insoluble, or difficultly soluble, by heat. But a higher heat than the temperature of the room is necessary to effect the solution of the ferric

hydroxide in the citric acid solution, and especially so when saturation is approached.

If 1 ml of ammonia water of ten per cent (H_3N) strength be added to the citric acid and water before the ferric hydroxide is added, the solution of the latter is greatly hastened and this small quantity of ammonia should not be objected to, amounting to only 1 Gm of H_3N in each liter of finished product.

A small amount of the ferric hydroxide remains undissolved, and is filtered away before the solution is evaporated to 100 Gm. This undissolved hydroxide is usually basic owing to the exposure of the normal hydroxide to heat.

Examinations made of many samples of solution of ferric citrate and of the ferric citrate in scales show that the percentage of iron in these preparations is quite variable. This is due to the varying degree of care observed in their manufacture, and especially to the conversion of a greater or less portion of the ferric hydroxide into insoluble meta-hydroxide. It is intended that the citric acid shall be saturated with ferric hydroxide, but the fact that a portion of the hydroxide remains undissolved at the end does not indicate saturation if the undissolved portion consists of basic ferric hydroxide for that is insoluble. Hence it is necessary in order to obtain as nearly uniform results as practicable, to adopt all the precautions described in the working directions given in the foregoing formula.

The Pharmacopœia states that the solution is of a strength corresponding to about 7.5 per cent of metallic iron. This is a minimum of iron. When well made the solution must contain about 35.5 per cent of normal ferric citrate, that being the proportion of ferric citrate formed by the quantity of citric acid employed. Assuming that the citric acid is all neutralized so as to form normal ferric citrate, the finished preparation contains about 8 per cent of iron. One hundred grams of the solution should, upon evaporation, yield about 42.5 per cent of scaled ferric citrate; but the yield depends in part upon how far the solution is evaporated before it is put on the glass plates to be dried, and the temperature at which the scaling is effected, or, in other words, the amount of moisture retained by the scaled salt. If the scaled salt correspond to $FeC_6H_5O_7.3H_2O$, the yield from 100 Gm of solution can not be over 42.46 Gm.

Description.—The solution of ferric citrate of the American Pharmacopœia is dark brown-red, odorless, and has a slightly ferruginous and only faintly acid taste. Its reaction on test-paper is acid. The sp. w. is not less than 1.250 at 15°.

IRON CITRATE WITH QUININE.

FERRI ET QUININAE CITRAS; U. S.

Ferric citrate	22 parts
Quinine, dried at 100° C. until it ceases to lose weight	3 parts

Dissolve the ferric citrate in 30 parts of distilled water in a porcelain dish over a water-bath at not over 60° C. Add the alkaloid and stir constantly until dissolved. Evaporate at the temperature named until the liquid is reduced to the consistence of syrup, and spread it on glass plates to dry in scales.

Keep the product in well stoppered bottles, in a cool, dark place.

Notes. This, the official citrate of iron and quinine of the United States, is reddish brown and only slowly, though perfectly, water soluble. The most commonly employed citrate of iron and quinine, however, is that prepared with the addition of ammonia, by which the preparation is not only rendered more readily soluble but changed also in color, being greenish yellow.

The quinine should be finely divided, and ought to be triturated first with five parts of water and then with a portion of cold solution of the citrate of iron, being afterwards well mixed with the whole before the digestion begins. Otherwise it may run together in lumps, which, afterwards, are very difficult to dissolve.

In this, as in all other scale salts of iron, it is necessary that no considerable excess of free citric acid should be contained in the preparation, for then the salt will not form scales, but adheres like a varnish to the glass plates.

"Quinine dried at 100° C., until it ceases to lose weight," is monohydrated quinine ($C_{20}H_{24}N_2O_2.H_2O$). The drying at this temperature is directed for the purpose of insuring uniformity.

Description.—Transparent scales, reddish to yellowish brown, slowly hygroscopic on exposure to moist air, odorless, having a bitter and mildly ferruginous taste, and slightly acid reaction. Slowly but wholly soluble in cold water, more readily so in hot water, and but partially soluble in alcohol.

Its solubility is impaired by age. It contains 12 per cent of mono-hydrated quinine.

German Preparation.

Citric acid	6 parts
Powdered iron	3 parts
Quinine	1 part

Dissolve the citric acid in 500 parts of water, heated in a porcelain dish; add the iron and digest over a water-bath, with frequent stirring, for 48 hours, or until the liquid acquires a reddish-brown color. Filter. Evaporate the filtrate to 50 parts, and then add the quinine previously triturated with a portion of the liquid. When the alkaloid has dissolved, evaporate to a syrupy consistence and spread it on glass plates to dry in scales.

Notes. This preparation contains both ferrous and ferric citrate. The quinine should be recently precipitated from a solution of 1.3 parts of quinine sulphate, dissolved in water with the aid of dilute sulphuric acid, the precipitant being sodium hydroxide.

Description.—Dull red-brown scales; odorless; bitter, ferruginous. Slowly but completely soluble in water. Contains from 9 to 10 per cent of quinine.

Swedish Preparation.

The working formula of the Swedish Pharmacopœia is as follows:

Citric acid	13 parts
Ferric chloride	12 parts
Quinine	3 parts
Ammonia water	57 parts

Dissolve the ferric chloride in 400 parts of cold water, and pour the solution into a mixture of the ammonia water and 60 parts

of water. Wash the ferric hydroxide thoroughly, and press out the moisture from it. Dissolve the citric acid in 25 parts of distilled water, warm the solution over a water-bath at not above 40° C. (104° F.), and add, during constant stirring, the ferric hydroxide in small portions at a time until the citric acid is saturated. Then add the quinine in the manner prescribed in the working formula for the preparation of the "Iron and Quinine Citrate" of the American Pharmacopœia.

Description.—Similar to the preparation of the American Pharmacopœia.

It contains about 14 per cent of quinine.

The Norwegian and Danish Pharmacopœias require from 10 to 12 per cent of quinine.

Soluble Citrate of Iron and Quinine.

FERRI ET QUININAE CITRAS SOLUBILIS; U. S.

Ferric citrate	85 Gm
Quinine, dried at 100° C. to a constant weight	12 Gm
Citric acid	3 Gm
Ammonia water.	
Distilled water, each sufficient.	

Heat 160 ml of distilled water in a porcelain dish over a water-bath to a temperature not exceeding 60° C., and add the ferric citrate, stirring until the scale-salt is dissolved. Triturate the quinine and the citric acid with 20 ml of distilled water, add this to the solution of ferric citrate in the porcelain dish, and stir constantly until all is dissolved. Now add gradually and with constant stirring about 50 ml of ammonia water, or a sufficient quantity to render the solution greenish-yellow, waiting after each addition of ammonia until the precipitated quinine shall have redissolved before another portion of the ammonia water is added. Evaporate the greenish-yellow solution over a water bath, at a temperature not exceeding 60° C. to a syrupy consistence and spread it on plates of glass or porcelain to dry in scales.

Keep the product in well-stoppered bottles protected from light.

Description.—Thin, transparent scales of a greenish golden-

yellow color; odorless; taste bitter, mildly ferruginous. Completely and quickly soluble in water. Only partially soluble in alcohol. Darkens on exposure to light. Hygroscopic in damp air.

It contains 12 per cent of monohydrated quinine.

British Preparation.

Solution of normal ferric sulphate, U. S..	328 ml
Sulphate of quinine.....	40 Gm
Diluted sulphuric acid.....	60 ml
Citric acid.....	123 Gm
Ammonia water, distilled water, each sufficient.	

Mix 320 ml of ammonia water with 1600 ml of cold water; add to this the solution of ferric sulphate, also previously diluted with 1600 ml of cold water, stirring constantly and briskly. Let the mixture stand for two hours, stirring it occasionally; then transfer it to a wetted muslin strainer, and when thoroughly drained, wash the hydroxide with water until the washings cease to give a precipitate with barium chloride.

Mix the quinine sulphate with 320 ml of water, add the diluted sulphuric acid, and, when the salt is dissolved, precipitate the quinine with a slight excess of ammonia water. Collect the precipitate on a filter, and wash it with 1000 ml of distilled water.

Dissolve the citric acid in 125 ml of distilled water, heated on a water-bath; add the ferric hydroxide, previously well drained; stir them together, and, when the hydroxide has dissolved, add the precipitated quinine, continuing the stirring until the alkaloid has also dissolved. Let the mixture cool. Add cautiously, a little at a time, 60 ml of ammonia water previously diluted with 80 ml of distilled water, stirring briskly, and allowing the quinine which separates with each addition of ammonia to re-dissolve before adding another portion. Filter the solution, evaporate it to the consistence of a thin syrup, and spread it on glass plates to dry in scales at not over 40° C.

Notes. The product is in greenish golden yellow scales, somewhat hygroscopic, and readily soluble in water. It contains about 16 per cent of quinine.

Solution of Citrate of Iron and Quinine.

Ammonio-ferric citrate	65 parts
Quinine, dried at 100° C. until it ceases to lose weight	12 parts
Citric acid	28 parts
Alcohol	30 parts

Dissolve the citrate of iron and ammonium in 200 parts of distilled water, in a tared porcelain dish; heat the solution over a water-bath to 60° C., add the citric acid, and, when this has dissolved, add the alkaloid previously triturated to a homogeneous mixture with a cooled portion of the liquid, and continue stirring until a perfect solution results. Evaporate to 160 parts, let cool, add the alcohol, and finally 10 parts of distilled water.

Should be kept in a well stoppered bottle, in a cool, dark place.

Description.—A dark greenish-yellow to yellowish-brown liquid. Odorless. Taste like that of citrate of iron and quinine. Reaction slightly acid. Corresponds to about 50 per cent of soluble citrate of iron and quinine.

IRON CITRATE WITH STRYCHNINE.

FERRI ET STRYCHNINAE CITRAS.

Citrate of iron and ammonium	98 parts
Strychnine	1 part
Citric acid	1 part

Dissolve the ammonio-ferric citrate in 100 parts of distilled water; and the alkaloid, together with the citric acid, in 20 parts of distilled water. Mix. Evaporate the mixture by water-bath at not over 60° C. to a syrupy liquid, and spread this on glass plates to dry in scales.

Keep the product in well stoppered bottles, in a cool, dark place.

Description.—Transparent, garnet-red scales, deliquescent on exposure to air, odorless, having a bitter and slightly ferruginous taste, and a slightly acid reaction. Readily and wholly soluble in water.

IRON (FERRIC) NITRATE SOLUTION.

LIQUOR FERRI NITRATIS.

An aqueous solution of ferric nitrate [$Fe(NO_3)_3$=242], containing about 6.2 per cent of the anhydrous salt, and corresponding to about 1.4 per cent of metallic iron.

Solution of ferric sulphate	18 parts
Ammonia water	15 parts
Nitric acid	71 parts
Distilled water.	
Water.	

Mix the ammonia water with 50 parts of cold water, and the solution of ferric sulphate with 150 parts of cold water. Add the latter solution slowly to the diluted ammonia water, with constant stirring. Let the mixture stand until the precipitate has subsided as far as practicable, and then decant the supernatant liquid. Add to the precipitate 100 parts of cold water, mix well, and again set the mixture aside, as before. Repeat the washing with successive portions of cold water, in the same manner, until the washings produce but a slight cloudiness with barium chloride test-solution. Pour the washed ferric hydroxide on a wet muslin strainer, and let it drain thoroughly. Then transfer it to a porcelain capsule, add the nitric acid, and stir with a glass rod, until a clear solution is obtained. Finally, add enough distilled water to make the finished product weight 100 parts. Filter, if necessary.

Reactions. $Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4\dot{N})_2SO_4$;

then, $Fe(OH)_3+3HNO_3=Fe(NO_3)_3+3H_2O$.

Description.—A clear, amber-colored or reddish liquid; odorless; taste acid and styptic. Reaction acid. Sp. w. 1.050 at 15°.

The solution of ferric nitrate of the British Pharmacopœia is more than twice as strong, containing about 3.01 per cent of iron. But the British formula employs an amount of nitric acid which corresponds to only 135 parts of absolute HNO_3 for each 1000 parts of finished solution, while the American formula employs nitric acid corresponding to 102 parts of absolute HNO_3 for each 1000 parts, so that while the British preparation contains more

than twice as much iron it uses only 33 per cent more nitric acid. Hence the British preparation contains both ferrous and ferric nitrate, while the American preparation contains only ferric nitrate.

The British Formula (1898)

is as follows:

Iron	20	Gm
Nitric acid (70% of HNO_3), 90 ml, or	127.8	Gm
Distilled water.		

Dilute the nitric acid with 320 ml of distilled water. Dissolve the iron in the mixture, being careful to moderate the reaction, should it become too violent, by the addition of a little more distilled water. Filter. Add enough distilled water to produce 600 ml of the solution (or 664.2 Gm).

This solution has the sp. w. 1.107.

IRON OXYCHLORIDE SOLUTION.

LIQUOR FERRI OXYCHLORIDI.

The following process is after the Swiss Pharmacopœia (1893):

Solution of ferric chloride, U. S.	300 parts
Ammonia water	400 parts
Hydrochloric acid	27 parts
Distilled water, sufficient.	

Warm the solution of ferric chloride to about 30° C. Dilute 300 parts of ammonia water with 300 parts of water. Add the ammoniacal liquid slowly and with constant stirring to the solution of ferric chloride. Then dilute the mixture with 1000 parts of distilled water.

Dilute 100 parts of ammonia water with 2000 parts of distilled water.

Now pour the iron mixture and the diluted ammonia, simultaneously, slowly, and with uninterrupted stirring, into 8000 parts of distilled water.

Wash the precipitate by the affusion and decantation of distilled water until the washings no longer give any reaction for chloride.

Collect the washed precipitate on a strainer and express the

water from it by strong pressure until the press cake is reduced to a weight not exceeding 400 parts.

Transfer this to a bottle, add the hydrochloric acid, and also enough distilled water to make the total weight of the mixture 1000 parts.

Let the mixture stand, shaking it occasionally, until the solid matter has dissolved.

Filter the solution.

The finished product should have the sp. w. 1.05.

Keep the product in a dark place, or in amber-colored bottles.

Description.—A clear brown-red liquid, containing an amount of ferric oxychloride corresponding to about 3.5 per cent of Fe. It should yield 5 per cent of ferric oxide on precipitation with ammonia in excess and ignition of the precipitate in the usual way.

Dialysed Solution of Iron.

LIQUOR FERRI DIALYSATUS.

Solution of ferric chloride	100 ml
Ammonia water	165 ml
Distilled water, sufficient.	

Mix 86 ml of the solution of ferric chloride with 500 ml of cold distilled water. Pour the mixture slowly and with constant stirring into the ammonia water previously diluted with 400 ml of cold distilled water. Wash the precipitate with cold distilled water first by decantation and afterwards on a wetted muslin strainer until the washings cease to be affected by test-solution of silver nitrate acidulated with nitric acid. Let drain, and then press out from the magma as much as possible of the moisture it holds.

Add the ferric hydroxide to the remainder of the solution of ferric chloride, stir well, and set aside until no more ferric hydroxide dissolves.

Decant the liquid from the undissolved portion, place it in a covered dialyser, and wash it by dialysis until the washings are nearly tasteless.

Then dilute the solution in the dialyser until it measures nearly 375 ml, and has a sp. gr. of 1.047.

Keep the product in tightly closed bottles and protected against light.

Notes. When 10 Gm of this solution is treated with ammonia water in excess, the precipitate, when washed, dried, and ignited, should weigh 0.50 Gm. A drop of the solution, diluted with water, should yield no precipitate with test-solution of silver nitrate acidulated with nitric acid.

This is the so-called "dialysed iron." It will be observed that the product is not the dialysed portion, but the residue after the dialysis. It contains a highly basic ferric oxychloride.

Description.—A dark reddish-brown liquid; odorless, and of a mild, non-styptic taste.

IRON PEPTONATE.

FERRUM PEPTONATUM.

Solution of oxychloride of iron (containing 3.5 per cent of Fe)	240 parts
Dried egg albumen	20 parts
Hydrochloric acid (25% of HCl)	34 parts
Pepsin	1 part
Distilled water, sufficient.	

Dissolve the egg albumen in 2000 parts of distilled water, to which have been previously added the pepsin together with 30 parts of the hydrochloric acid. Let this mixture stand, stirring or shaking it occasionally, for a period of 12 hours in a place where the temperature is about 40° C. Then let the solution cool to 20° C., filter or strain it, and carefully neutralize it with a very weak solution of sodium hydroxide. Should a precipitate be formed, filter the liquid.

Dilute the solution of ferric oxychloride with 2000 parts of distilled water, and add this solution to the solution of digested egg albumen. Mix well.

Neutralize the mixture carefully with a very weak solution of sodium hydroxide, stirring well.

Wash the precipitated iron peptonate with distilled water as long as the washings give a *precipitate* with silver nitrate solu-

tion. [The washing should not be continued while mere opalescence is caused by the silver solution.]

Collect the precipitate on a cloth strainer, let it drain well, transfer it to a porcelain dish, add the remaining 3 parts of the hydrochloric acid, and heat the mixture carefully over a water-bath, at not over 56° C., until solution is effected.

Evaporate the liquid at not above 50° C. to the consistence of syrup, spread the thick liquid upon glass plates, and let it dry in scales.

Description.—Red-brown scales, odorless, and of feebly bitterish ferruginous taste. Forms a clear solution with water. The solution has an acid reaction. It should not become turbid on heating above 60° C. or on addition of alcohol (absence of albumen).

Solution of Iron Peptonate

may be prepared by mixing the still moist iron peptonate made from 20 parts of dry egg albumen with 1780 parts of distilled water and 200 parts of brandy.

Should be protected against light.

Solution of Iron and Manganese Peptonate.

1. Make a solution of 10 parts of citric acid in 50 parts of distilled water, and add enough ammonia water (24.2 parts) to make normal ammonium citrate.

2. Dissolve 3.7 parts of manganous chloride in 10 parts of distilled water.

3. Dissolve 24 parts of iron peptonate in 300 parts of distilled water.

4. Mix 150 parts of alcohol with 400 parts of distilled water.

Add solution 1 to 2. Then add 3. Then 4. Finally add enough distilled water to make the total product weigh 1000 parts.

Notes. This preparation is usually flavored with aromatic tincture (Swiss Ph.), and the tinctures of cinnamon and vanilla, together with some acetic ether.

Must be protected against light.

Should it become unclear it may be rendered clear again by gently warming it, and then adding a few drops of ammonia water.

IRON AMMONIUM TARTRATE.

FERRI ET AMMONII TARTRAS.

(Ferryl-Ammonium Tartrate.)

Solution of normal ferric sulphate.....	90 parts
Tartaric acid	18 parts
Ammonia water	72 parts
Ammonia water, distilled water, each sufficient.	

Mix the ammonia water with 180 parts of cold water; add gradually, and with constant stirring, the solution of tersulphate of iron previously diluted with 900 parts of cold water.

Pour the mixture on a wetted muslin strainer, allow the magma to drain, and then return it to the precipitation vessel and mix it well with 1000 parts of cold water. Drain it again on the strainer, and repeat the operation once, or oftener, until the washings only give a slight cloudiness with barium chloride solution. Then allow it to drain thoroughly. Dissolve one-half of the tartaric acid in 45 parts of distilled water, neutralize the acid exactly with ammonia water, then add the remainder of the tartaric acid, and dissolve it by the aid of heat. Keep the solution of ammonium bitartrate hot, but not above 60° C., over a water-bath, and add to it, with constant stirring, the ferric hydroxide, in small portions at a time, until no more will dissolve. Filter the solution while hot, evaporate it at not over 60° C. to the consistence of syrup, and spread it on glass plates to dry in scales.

Keep the product in amber-colored bottles, tightly corked, and put in a cool, dark place.

Notes. The remarks under Iron and Potassium Tartrate apply in a general way to this process as well. The ammonium bitartrate is $H_4NHC_4H_4O_6$, by which the ferric hydroxide is dissolved in a manner analogous to the solution.effected by digesting the hydroxide with potassium bitartrate in making the tartrate of iron and potassium.

Description.—Transparent, garnet-red to brown-red scales, somewhat lighter and clearer in color than the tartrate of iron and potassium. Odorless. Taste sweetish, slightly ferruginous. Very

soluble in water. Insoluble in alcohol. Produces more clear and permanent solutions than potassium iron tartrate.

IRON POTASSIUM TARTRATE.

FERRI ET POTASSII TARTRAS.

(Ferryl-Potassium Tartrate.)

Solution of normal ferric sulphate.....	12 parts
Potassium bitartrate	4 parts
Distilled water.	
Ammonia water, sufficient.	

Mix 10 parts of ammonia water with 20 parts of cold water; add gradually, and with constant stirring, the solution of tersulphate of iron, previously diluted with 100 parts of cold water. Pour the mixture on a wet muslin strainer, allow the magma to drain, and then return it to the precipitation vessel and mix it well with 100 parts of cold water. Drain it again on the strainer, and repeat this operation once or twice, as may be necessary, until the washings produce but a slight cloudiness with test-solution of barium chloride. Let the magma be well drained; then forcibly press out of it as much of the water as possible. Add it in small portions at a time to a mixture of the distilled water and the potassium bitartrate, heated over a water-bath at not over 60° C., stirring constantly until all the ferric hydroxide has been added and dissolved. Filter while hot and set the filtrate aside in a cool, dark place for twenty-four hours; then stir it well with a porcelain or glass stirrer until any precipitate which may have formed in the liquid is evenly distributed through it. Then cautiously add just enough ammonia water to dissolve this precipitate, filter, and evaporate the solution in a porcelain dish to the consistence of thick syrup, and spread it on glass plates to dry in scales.

Keep the product in well corked amber bottles, in a dark place.

Reaction. Ferric hydroxide is first prepared from the ferric sulphate:

$$Fe_2(SO_4)_3+6H_4NOH=Fe_2(OH)_6+3(H_4N)_2SO_4.$$

The ferric hydroxide is then dissolved by the potassium tartrate, forming several compounds, one of which is perhaps:

$$3KHC_4H_4O_6+Fe(OH)_3=K_3Fe(C_4H_4O_6)_3+3H_2O,$$

while another is probably made in this way:

$$KHC_4H_4O_6+Fe(OH)_3=K(FeO)C_4H_4O_6+2H_2O.$$

But the tartrate of iron and potassium probably is not a definite double salt; the pharmacopœial products are all variable as to the proportion of iron.

Notes. Whether prepared from solution of tersulphate of iron or from solution of chloride of iron, the ferric hydroxide must be carefully precipitated, from cold dilute solutions, and well washed. When made from chloride it is more readily washed clean. The hydroxide does not dissolve rapidly in the liquid, or the soluble tartrate of iron and potassium is but slowly formed from the ferric hydroxide and the potassium bitartrate. Several hours are required to complete the solution. In view of the fact that ferric hydroxide is so liable to become basic and insoluble when subjected to heat, it is best to add the ferric hydroxide in portions, to regulate the heat carefully and to stir constantly. The success of the whole process depends to a great extent upon the character of the ferric hydroxide and upon the precautions described in the foregoing. The addition of ammonia is intended to render the product readily water soluble without residue. The Pharmacopœia does not direct that the solution should be filtered, but this is necessary to obtain a good product.

Description.—Transparent garnet-red or brown-red scales, odorless, taste sweetish, slightly ferruginous. Very soluble in water; insoluble in alcohol.

Impure Malate of Iron.

Iron wire, cut	20 Gm
Crab apples, nearly ripe	500 Gm

Pare the apples, beat them into a pulp, and express the juice. Add the iron, mix well, set the mixture in a warm place for ten days, or until all signs of effervescence cease, stirring frequently. Replace from time to time the water lost by evaporation. Heat the mixture on a water-bath at 70° C. for six hours, replacing water lost by evaporation. Add 100 ml of warm water, express,

mix the residue with another 100 ml of warm water, and express again. Mix the liquids and set the mixture aside in a cool place to settle. Decant the clear liquid, or filter if necessary, and evaporate to a thick extract.

The product is greenish black, has a styptic taste, yields a clear solution with water, and usually contains about 7 to 8 per cent of iron.

Note. Any sour apples may be used if wild apples are not available. This preparation is still retained in several pharmacopœias, and tinctures are made from it.

Description.—A dark brown-red (nearly black) extract-like mass.

IRON (FERRIC) FERROCYANIDE.

FERRI FERROCYANIDUM.

(Prussian Blue.)

$Fe_4(FeCy_6)_3$=860.

Solution of ferric chloride, U. S. P..... 23 ml
Potassium ferrocyanide ..... 14 Gm
Distilled water, sufficient.

Dilute the solution of ferric chloride with 450 ml of distilled water. Dissolve the potassium ferrocyanide in 300 ml of distilled water. Filter both liquids. Add the solution of potassium ferrocyanide slowly and with constant stirring, to the solution of chloride of iron. Let the precipitate settle, and wash it first by decantation, changing the wash water as frequently as practicable; then transfer the magma to a wetted muslin strainer and continue the washing until the washings are tasteless. Let drain, and dry it on the muslin, spread in a thin layer. If necessary, finish the drying on glass plates in the drying chamber. Reduce the product to a fine powder.

Reaction. $3K_4FeCy_6+4FeCl_3=12KCl+Fe_4(FeCy_6)_3$.

Notes. The dilute condition of the solutions is necessary because the precipitate is very voluminous. It is also necessary that the ferric chloride should be in excess over the potassium ferrocyanide

throughout the reaction, that for this purpose the cyanide should be added to the chloride and not *vice versa*, and that some ferric chloride should remain undecomposed at the end. Otherwise the product will be contaminated with more potassium salt than when the directions given are observed. Under any circumstances there will be a slight amount of potassium compound in the product. Any attempt to wash out all the potassium salt will result in the formation of ferric oxide, which remains in the preparation.

The precipitate is a voluminous magma which is difficult to wash, as it settles very slowly. Washing on a filter is impracticable, because the precipitate completely closes the pores of the paper.

The presence of a little free HCl in the liquid (which will be insured by using the solution of ferric chloride of the U. S. P.) considerably facilitates the washing.

IRON (FERRIC) HYDROXIDE.

FERRI HYDROXIDUM.

$Fe(OH)_3 = 107$.

Solution of normal ferric sulphate (50 Gm, or)	38 ml
Ammonia water (40 Gm, or)	42 ml
Water, sufficient.	

Dilute the ammonia water with 100 ml of cold water in a precipitation vessel of about one liter's capacity. Dilute the solution of ferric sulphate with 500 ml of cold water, and pour this into the dilute ammonia slowly and during constant stirring.

Wash the precipitated ferric hydroxide with cold water, first by affusion and decantation, and afterwards on a wetted muslin strainer, until the washings are tasteless. Then let the magma drain, return it to the precipitation vessel, and mix it well with as much cold water as the vessel will hold, or about one liter. Let settle, decant the supernatant liquid, and again transfer the magma to the wetted strainer and allow it to drain. Continue the washing in this manner until the wash water is no longer affected by test-solution of barium chloride.

[When the ferric hydroxide is to be used as an antidote for

arsenic, the whole process must be hastened, and the washing need not be carried farther than to remove the mother liquor. To effect this the precipitate is allowed to drain until most of the liquid has run off, and the strainer is then gathered up so as to enclose the magma, which is then forcibly pressed with the hands until no more liquid can be squeezed out, after which enough water is added to the hydroxide to make the weight of the whole product equal to twice the weight of the solution of ferric sulphate used.]

Reaction. $Fe_2(SO_4)_3+6H_4NOH=2Fe(OH)_3+3(H_4N)_2SO_4$.

Notes. *Dilute* and *cold* solutions are necessary. The diluted solution of ferric sulphate should be poured slowly, or in a small stream, into the diluted ammonia water during constant stirring, as directed. When the whole of the iron solution has been added, the mixture should still have a decidedly ammoniacal odor; as the ammonia gas is diffused through the stratum of air above the surface of the liquid, that air must be fanned or blown away before the odor of the liquid itself can be ascertained.

There are several ferric hydroxides, differing from each other in the proportions of HO they contain, in color, and in their solubility in acids. The product intended by the Pharmacopœia is $Fe(OH)_3$; another ferric hydroxide is $Fe_2O(OH)_4$; and dried ferric hydroxide is $OFe(OH)$. The more basic it is, and the less water it contains, the less readily soluble is it. As obtained by the official process, all the necessary precautions being observed, the product is dark reddish-brown and readily soluble in citric acid, and in glacial acetic acid. When yellowish-brown, brown, grayish-brown, or clay-colored, it is basic, insoluble and unfit for the uses for which it is intended in pharmacy.

The solution of ferric sulphate must contain the full amount of ferric sulphate, and be free from ferrous salt in order that the quantity of ferric hydroxide obtained from it may be definite; the importance of this will be readily understood from the fact that the proportions of the materials prescribed in several working formulas for ferric salts are calculated with reference to the amount of ferric hydroxide theoretically yielded by the solution of ferric sulphate.

In summer it is best to use ice for cooling the dilute solutions previous to the precipitation. The chemical reaction generates heat, and if the solutions are already warm the temperature of

the mixture is liable to rise sufficiently high to cause the formation of light-colored meta-hydroxide. But the liquids must not be ice-cold.

The official solution of ferric sulphate, containing 27.8 per cent of $Fe_2(SO_4)_3$, yields 14.84 per cent of ferric hydroxide. The official solution of ferric chloride, containing 38.7 per cent of $FeCl_3$, yields about 24.92 per cent of $Fe(OH)_3$.

Should the quantity of ammonia used be insufficient, or should it be added to the ferric sulphate instead of *vice versa,* basic ferric salts will be formed. If the H_3N is added to the iron solution a dark red-brown solution containing ferric salt is obtained at first, and no ferric hydroxide is thrown down until more ammonia is added.

Ferric hydroxide may also be precipitated from ferric chloride or ferric nitrate, and soda may be used as a precipitant instead of ammonia.

If solution of ferric chloride is used the hydroxide formed is more easily washed free from the ammonium salts. If ferric nitrate is used, the product is soluble in an excess of potassium carbonate (Stahl's tincture).

Ferric hydroxide precipitated with soda instead of ammonia is lighter in color and extremely difficult to wash free from sodium salt.

Ferric hydroxide forms very soluble compounds, with sugar and with glycerin. It is also soluble in solutions of ferric salts, as in solution of ferric chloride. The precipitation of ferric hydroxide from solutions of ferric salts by means of alkalies is prevented by the presence of citric acid, tartaric acid, sugar, glycerin, and certain other organic substances.

Description.—When moist it is a dark red-brown magma, insoluble in water, but soluble in citric, tartaric, acetic, hydrochloric, sulphuric, and nitric acids. When dried it is a reddish-brown amorphous powder, less readily soluble in acids than the moist hydroxide.

Uses. For the preparation of ferric acetate, citrate, tartrate, nitrate, etc. Also as an antidote for arsenic, with which it forms insoluble arsenate:

$$4Fe(OH)_3+As_2O_3=Fe_3(AsO_4)_2+Fe(OH)_2+5H_2O.$$

For whatever purpose it may be required it must be freshly prepared, as it cannot be preserved from decomposition by which meta-hydroxides are formed, which in no case can take the place of the freshly precipitated ferric hydroxide.

Iron (Ferric) Hydroxide with Magnesia.

FERRI HYDROXIDUM CUM MAGNESIA.

[Antidote for Arsenic.]

Solution of normal ferric sulphate (65 Gm, or)	50 ml
Magnesia	10 Gm

Mix the solution of tersulphate of iron with 130 ml of water.

Stir the magnesia with about 150 ml of water to a thin milky mixture, transfer this at once to a wide-mouthed bottle of two liters' capacity, and add one liter of water.

If the magnesium oxide mixed with 15 times its weight of water be allowed to stand too long before the remainder of the water is added it will form a firm gelatinous mass of magnesium hydroxide.

The two liquids should be kept in a cool place, and as these materials are intended exclusively for the preparation of an effective antidote for arsenical poisoning, the particular place where they are to be found must be well known by every one concerned.

When the antidote is wanted, the solution of ferric sulphate is at once poured into the magnesia milk, and the mixture thoroughly shaken. It is then ready for immediate use.

The preparation contains ferric hydroxide, magnesium hydroxide and magnesium sulphate.

Another Formula.

"Antidote for arsenic" is prepared as follows, according to the Pharmacopœia of the Netherlands:

Ferric chloride	90 Gm
Distilled water	550 ml

Make a solution, and keep it in a bottle.

Magnesia	28 Gm
Distilled water	520 Gm

Mix well and keep this mixture in another bottle.

When required for use mix equal volumes of the two liquids and shake well.

IRON (FERRIC) HYPOPHOSPHITE.

FERRI HYPOPHOSPHIS.

$Fe(PO_2H_2)_3=251.$

Sulphate of iron and ammonium	30 parts
Sodium hypophosphite	20 parts

Dissolve each salt in 150 parts of water, and filter the solutions. Add the solution of the iron alum to that of the sodium hypophosphite, stirring constantly. Set aside for a few hours. Transfer the precipitate to a wetted filter or muslin strainer, drain it thoroughly, and wash it with 150 parts of cold distilled water. Drain, press out as much as possible of the moisture from the magma, and dry the product between blotting paper with the aid of gentle heat, protecting the preparation as far as practicable from the air.

Reaction. $2(FeH_4N(SO_4)_2.12H_2O)+6(NaPO_2H_2.H_2O)$

$=2Fe(PO_2H_2)_3+(H_4N)_2SO_4+3Na_2SO_4+30H_2O.$

Notes. The quantity of water used is necessarily limited to prevent loss, because ferric hypophosphite is not quite insoluble in water, and if the washing should be done with the large quantities of water required to thoroughly remove the sulphates of the mother liquor from the bulky magma of hypophosphite, considerable loss must follow, if not the disappearance of the entire product. The precipitate must, therefore, be carefully freed from all mother liquor that can be removed by draining it before it is washed, and the quantity of water prescribed for washing it should not be exceeded.

Ferric hypophosphite is soluble in a strong, hot solution of citrates of potassium and sodium.

Description.—A grayish powder, odorless and almost tasteless,

slightly soluble in water, readily soluble in acetic acid, and in solutions of potassium citrate or of sodium citrate.

IRON (FERROUS) IODIDE.

FERRI IODIDUM.

FeI_2=309.

Iron wire, cut	6 parts
Iodine	17 parts
Distilled water	20 parts

Digest in a flask at a moderate heat until all odor of iodine has ceased and a green solution has been obtained. Filter the solution and evaporate the filtrate as rapidly as possible in a polished iron dish until a trial drop solidifies on cooling. Then pour the liquid out on a porcelain slab, and as soon as it has hardened break it into pieces while still warm, and put it into small, dry, warm bottles, which must be at once tightly stoppered.

For reaction and notes see Syrup of Ferrous Iodide.

Description.—Ferrous iodide is a crystalline solid, which, in mass, appears nearly black. In solution it is green. But the product easily decomposes, and it is for that reason scarcely employed except in the form of "saccharated iodide of iron," or as "syrup of ferrous iodide."

Saccharated Ferrous Iodide.

FERRI IODIDUM SACCHARATUM.

Iron, in the form of fine, bright wire, cut into small pieces	6 parts
Reduced iron	1 part
Iodine	17 parts
Distilled water.	
Dry milk sugar, of each, sufficient.	

Mix the iron wire, iodine and 20 parts of distilled water in a flask, and shake them together occasionally until the reaction ceases, and the liquid has acquired a green color and lost the odor of iodine. Warm the flask and contents, if necessary, to complete the reaction. Filter the liquid through a small white paper

filter previously wetted with distilled water, into a porcelain dish containing about 40 parts of powdered milk sugar. Rinse the flask and the undissolved iron wire with a little distilled water, and pass the rinsings through the same filter as before used into the porcelain dish.

Mix the contents of the dish by stirring with a glass rod or porcelain spatula. Evaporate the water from the mixture by water-bath heat, stirring frequently, until a dry mass remains. Transfer the dry residue while still hot to a clean, bright iron mortar heated by placing it in boiling water and then wiped dry, powder it, add the reduced iron and enough powdered milk sugar to make the weight of the total product 100 parts, and triturate these ingredients together until intimately mixed and reduced to a fine powder.

Transfer the powder at once to small, warm, perfectly dry bottles, which should be filled, and tightly stoppered.

Notes. See the reaction and remarks given under the title of Syrup of Ferrous Iodide.

The ferrous iodide contained in this preparation is protected from decomposition by the milk sugar and the reduced iron. It should contain at least 20 per cent of ferrous iodide.

Description.—A yellowish-white, grayish-white, or greenish-gray powder; odorless; hygroscopic; having a sweetish, ferruginous taste. Soluble in 7 parts of water at 15°.

Syrup of Ferrous Iodide.

SYRUPUS FERRI IODIDI.

A syrupy liquid containing 10 per cent of ferrous iodide (FeI_2, 309).

Iron, in the form of fine wire, and cut into small pieces	25 Gm
Iodine	83 Gm
Sugar, in coarse powder	600 Gm
Distilled water, sufficient.	

Introduce the iron into a flask, add 200 ml of distilled water, and afterward the iodine. Shake the mixture occasionally, until the reaction ceases and the solution has acquired a green color and

has lost the odor of iodine. Place the sugar in a porcelain dish and filter the solution of ferrous iodide into the sugar. Rinse the flask and iron wire with 90 ml of distilled water, and pass the washings through the filter into the sugar. Stir the mixture with a glass rod, heat it to the boiling point on a sand-bath, and having filtered the syrup through white paper into a tared bottle, add enough distilled water to make the product weigh 1000 Gm. Lastly, shake the bottle, which should be completely filled and securely stoppered.

Reaction. $2Fe+2I_2=2FeI_2$.

Notes. The proportion of iron combining with 8.20 parts of iodine to form ferrous iodide is only a little over 1.80 parts, so that the iron is present in large excess. This is to facilitate the complete saturation of the iodine. At first the reaction is tardy, and may require the aid of heat to start it; but as soon as some ferrous iodide has been formed the iodine dissolves in the solution, and after that the liquid acts rapidly on the iron with the evolution of heat. If this elevation of temperature is too great there will be a loss of iodine by vaporization. The heat must not be so great that violet vapors appear in the flask. It is safest to add the iodine in small portions at a time, during brisk stirring, waiting for the liquid to become green instead of red-brown after each addition before adding more.

The reaction between iron and iodine will take place at all temteratures, but is slow in cold. Should the reaction proceed at a comparatively low temperature until all the iodine has been added, and cease without leaving a liquid of bright green color when filtered, the application of heat before removing the undissolved iron will always bring out the green color, which, together with the absence of iodine odor, is the sign of completed reaction.

To prevent oxidization the solution of ferrous iodide should be filtered as rapidly as possible and while still hot, directly into the sugar.

Success in preparing a perfect syrup of iodide of iron depends upon a perfectly completed reaction, careful control of the temperature so that no iodine shall be lost, the use of pure distilled water and pure white sugar, and scrupulous cleanliness in regard to the vessels and implements used.

Syrup of iodide of iron may be successfully preserved in corked

bottles, but with much less risk in glass-stoppered bottles. Exposure to light seems to decolorize the preparation, even after the green color has changed to a pale yellowish. But in order to best preserve a bright green syrup of ferrous iodide from change it should be kept in a shaded place.

Long exposure to light almost completely decolorizes the preparation, and the sugar in the syrup becomes to a great extent inverted (J. H. Long).

Unless the bottle containing the syrup is filled, the surface of the preparation soon becomes yellow or yellowish-brown from oxidation by contact with the air above it. Hence the direction that the bottle shall be filled.

When this preparation becomes slightly discolored—yellowish-green but not yellow or yellowish-brown—the bright green color can usually be restored by heating it to near the boiling point in a flask by means of a water-bath.

Straining does not suffice to make the product clear. It must be filtered through white filter-paper.

As the preparation is prone to discoloration by decomposition, assuming a yellowish-brown color from free iodine, various means have been suggested to prevent this change. It was at one time proposed to place a piece of polished iron—for example, a nail filed until bright over its whole surface—in the syrup; but this is worse than useless. Another and more successful plan was to add tartaric or citric acid, in the proportion of about 1 part in 500. All such additions are, however, unnecessary, as the preparation will keep well if properly made and put into clean, glass-stoppered bottles in the manner indicated above.

Description.—Must be perfectly clear pale green, odorless, with a sweet strongly ferruginous taste. Reaction neutral. Sp. w. about 1.353 at 15°.

The size of the container should be determined by the rate at which the preparation is dispensed in the pharmacy. It should not be so large that the contents may not be entirely consumed in a few weeks. Bottles holding 500 Gm are as large as should be used, and bottles containing 100 Gm are not too small when the demand for the preparation is limited.

The same preparation (containing 10 per cent of ferrous iodide) is contained in all other pharmacopœias.

Glycerite of Ferrous Iodide.

Iodine	83 Gm
Iron wire	25 Gm
Distilled water	150 ml
Glycerin	300 ml

Digest the iron wire and iodine together in the water, contained in a flask, until the liquid has acquired a bright green color, and lost all odor of iodine. Filter the solution into the glycerin, mix well, and evaporate the mixture until it weighs 500 Gm.

Notes. This glycerite keeps indefinitely, mixes clear with water, alcohol, and syrup, and is exactly twice the strength (in ferrous iodide) of the Pharmacopœial syrup of iodide of iron (U. S.).

IRON (FERROUS) LACTATE.

FERRI LACTAS.

$$Fe(C_3H_5O_3)_2.3H_2O=288.$$

Whey	1000 ml
Milk sugar	50 Gm
Sodium bicarbonate	60 Gm
Ferrous sulphate	40 Gm
The white of two eggs.	
Diluted sulphuric acid.	
Water.	

Add the milk sugar to one liter of the clear whey obtained from spontaneously soured skimmed milk, in a cylindrical vessel of about two cubic decimeters capacity. Let the mixture stand in a place where the temperature is maintained at between 30° and 45°. After fermentation has commenced (within a day or two) and the liquid is sour from lactic acid, neutralize with a portion of the sodium bicarbonate. Repeat this neutralization every day or two, adding each time enough sodium bicarbonate to completely neutralize the lactic acid formed.

When the mixture no longer acquires a decidedly acid reaction within three or four days after neutralization, and when about 60 Gm of sodium carbonate has been consumed, add enough di-

luted sulphuric acid to render the liquid distinctly acid. Then add the whites of two eggs. Mix thoroughly.

Now heat the mixture to boiling. Filter the hot liquid. Evaporate the filtrate to about 250 ml.

Dissolve the ferrous sulphate in 80 ml of hot distilled water, and filter. Add this solution while hot to the hot solution of sodium lactate. Remove from the hot mixture the brown flocculent precipitate formed, passing the liquid through a flannel strainer. Set the hot colature aside in a cold place for two days. Collect the crystallized ferrous lactate on a strainer. Let the crystalline mass be well drained. Wash it with a little cold distilled water, and afterwards with a little alcohol. Press the moist lactate between blotting paper, changing the paper several times. Dry the product well with the aid of moderate heat.

Keep the product in small tightly closed bottles in a cool, dry place.

Reactions.

$C_{12}H_{22}O_{11}+H_2O=4HC_3H_5O_3$; then

$HC_3H_5O_3+NaHCO_3=NaC_3H_5O_3+H_2O+CO_2$; and, finally,

$2NaC_3H_5O_3+FeSO_4=Fe(C_3H_5O_3)_2+Na_2SO_4$.

Notes. The whey contains 4 to 5 per cent of milk-sugar, and this, together with that added, yields lactic acid, as shown above.

Unless this acid is neutralized from time to time, the lactic fermentation, which ordinarily continues for a week, may change to vinous and acetic fermentation. The production of alcohol and acetic acid might also follow if the temperature of the liquid is below 20° C.

$$C_{12}H_{22}O_{11}+H_2O=4C_2H_5OH+4CO_2.$$

Should the temperature be allowed to exceed 40° C., or an insufficient quantity of milk-sugar be used, butyric acid will be formed.

$$2HC_3H_5O_3=HC_4H_7O_2+3CO_2+2H_2.$$

To arrest further fermentation, the liquid is heated to the boiling point, which at the same time coagulates, and facilitates the

removal of albuminoid matters. Dilute sulphuric acid is added to prevent the formation of ferric salt, throwing down ferric hydrate, which is filtered out, and to neutralize any alkali which may have been added in excess.

The crystals of ferrous lactate are removed from the mother liquor, and then rinsed free from sodium sulphate by cold water, after which the water adhering to the salt is washed off with a little alcohol to facilitate subsequent drying. The product must be dried at not above 30° to 40° as rapidly as practicable to prevent oxidation.

If not thoroughly dry when put up it soon oxidizes.

A preparation entirely free from ferric lactate cannot be produced. The less ferric salt the preparation contains, the greener will be its color. It should have the proper greenish color, and yield a greenish solution with water. As the solution of this salt in water is always a slow process, the ferrous lactate should be triturated to fine powder before adding the water.

Another Method.

Crystallized calcium lactate	500 Gm
Hydrochloric acid	320 ml
Iron wire	100 Gm
Distilled water, sufficient.	

Put the iron into a mixture of the acid with 200 ml of water contained in a flask. When effervescence has ceased, heat to the boiling point. Let cool. Filter the solution. Add enough water to make the solution measure 600 ml.

Dissolve the crystallized calcium lactate in 2 liters of boiling distilled water.

Mix the solutions and set the mixture aside in a cool place for two or three days.

Collect the ferrous lactate on a filter, wash it with a little alcohol, and dry it at a temperature not exceeding 50° C.

Reaction. $FeCl_2+Ca(C_3H_5O_3)_2=Fe(C_3H_5O_3)_2+CaCl_2$.

Notes. An additional amount of ferrous lactate may be recovered from the mother liquor by adding alcohol to it and allowing it to stand a few hours, ferrous lactate being almost insoluble in alcohol while calcium chloride is soluble in it.

Description.—Yellowish green crystalline masses, or a greenish white powder, nearly inodorous, having a sweetish ferruginous taste, and a slightly acid reaction. Soluble in 40 parts of water at 15° C., and in 12 parts of boiling water. Freely soluble with a green color in solutions of alkali citrates. Nearly insoluble in alcohol.

IRON (FERRIC) OLEATE.

FERRI OLEAS.

$$Fe(C_{18}H_{33}O_2)_3=899.$$

Solution of normal ferric sulphate.....	90 ml
White castile soap, in fine powder.....	150 Gm

Dilute the solution of ferric sulphate with 5,000 ml of water, and dissolve the soap in 2,500 ml of hot water. Mix the solutions. Wash the precipitated oleate twice with hot water, using about ten liters each time.

Reaction.

$$Fe_2(SO_4)_3+6NaC_{18}H_{33}O_2=2Fe(C_{18}H_{33}O_2)_3+3Na_2SO_4.$$

Notes. The yield is about 120 Gm.

Ferrous oleate can also be made by double decomposition, from ferrous sulphate and soap, but the greenish ferrous oleate soon oxidizes.

Description.—Ferric oleate is a dark red plaster-like solid. Soluble in oleic acid and in fixed oils.

IRON (FERROUS) OXALATE.

FERRI OXALAS.

$$FeC_2O_4.H_2O=162.$$

Ferrous sulphate.....	330 Gm
Oxalic acid.....	150 Gm
Ammonia water	300 ml

Dissolve the oxalic acid in the ammonia water diluted with 2,000 ml of water; and the ferrous sulphate in 3,000 ml of hot

water; filter the solutions, and then mix them. Wash the precipitate by decantation and afterwards on a filter until the washings are tasteless. Dry the product between bibulous paper, with the aid of gentle heat.

Reaction.

$$FeSO_4+(H_4N)_2C_2O_4=FeC_2O_4+(H_4N)_2SO_4.$$

Notes. Ferrous oxalate can also be made by the process official in the U. S. P. of 1870, which prescribed ferrous sulphate dissolved in water to be added to a solution of oxalic acid. When this process is followed ferrous oxalate and free sulphuric acid are formed, the precipitate separates slowly, and a considerable loss results from the fact that the oxalate is not insoluble in the sulphuric acid. By neutralizing nearly all of the oxalic acid with ammonia, using a slight excess of oxalic acid, and mixing the solution of ferrous sulphate with the acid solution of ammonium oxalate, the precipitate falls at once, and no loss of product is sustained. If a perfectly neutral solution of ammonium oxalate is used, the product will have a dull reddish yellow color.

To obtain a bright yellow ferrous oxalate the mother liquor in which the precipitate is formed must be decidedly acid.

Description.—A light-yellow crystalline powder; odorless; nearly tasteless. Nearly insoluble in water, and insoluble in alcohol.

IRON (FERRIC) OXIDE.

FERRI OXIDUM.

$$Fe_2O_3=160.$$

Heat dried ferric hydroxide strongly until it ceases to lose weight.

$$2Fe(OH)_3=Fe_2O_3+3H_2O.$$

Seven parts of dry ferric hydroxide will yield rather more than five parts of oxide. It is brown-red.

Another Method.

Heat ferrous oxalate until completely decomposed.

$$4FeC_2O_4+3O_2=2Fe_2O_3+8CO_2.$$

The product obtained from ferrous oxalate is extremely finely divided and therefore soft and of a rich, dark-brown color. This is used by jewellers to polish gold.

Magnetic Oxide of Iron.

$$Fe_3O_4=232.$$

Solution of ferric sulphate, U. S.....	230 ml
Ferrous sulphate.....	60 Gm
Ammonia water (10% of H_3N).....	500 ml.
Water.	

Dissolve the ferrous sulphate in two liters of water and filter. Add the solution of ferric sulphate.

Dilute the ammonia water with one liter of water.

Pour the iron solution gradually into the diluted ammonia water, stirring well.

Set the mixture aside to settle. Decant the supernatant liquid from the precipitate. Then add enough water to make the whole mixture measure about three liters.

Boil this mixture about fifteen minutes, or until the brown ferroso-ferric hydroxide is changed to the nearly black ferroso-ferric oxide.

Wash the precipitate, first by affusion and decantation of hot water, and afterwards on a filter, until the washings cease to give a precipitate with test-solution of barium chloride. Then dry the product with the aid of heat.

Reaction.

$$Fe_2(SO_4)_3+FeSO_4+8H_4NOH$$

$$=Fe_2O_3.FeO+4(H_4N)_2SO_4+4H_2O.$$

Description.—A fine, heavy, brownish-black, odorless and tasteless powder.

Another Method.

Ferrous sulphate	300 parts
Sulphuric acid	39 parts
Nitric acid	28 parts
Ammonia water	640 parts
Distilled water, sufficient.	

Add the sulphuric acid slowly to 200 parts of distilled water in a porcelain dish, stirring constantly. Heat the mixture to a temperature of over 90° C. on a water-bath. Add the nitric acid and mix well. Then add the ferrous sulphate gradually, stirring constantly, waiting after each addition until effervescence ceases before adding more. When two-thirds of the ferrous sulphate has been added, the remaining 100 Gm of that salt may be added at once, together with 500 parts of distilled water. When the salt has completely dissolved, filter the solution; then add 10,000 parts of distilled water.

Mix the ammonia water with 3,000 parts of distilled water.

Pour the iron solution slowly into the dilute ammonia solution, stirring constantly.

When the precipitate has subsided, decant the supernatant liquid, transfer the ferroso-ferric hydroxide to a porcelain dish, add an equal volume of water, and boil the mixture about fifteen minutes or until the color of the precipitate is changed from brown to black. Wash the ferroso-ferric oxide with boiling water until the washings are no longer affected by barium nitrate test solution. Let the oxide drain, press out from it as much of the water as it is practicable to remove by that means, and dry the product as rapidly as practicable.

When perfectly dry, keep it in well-stoppered bottles.

IRON PHOSPHATE; BLUE.

FERRI PHOSPHAS COERULEUS.

[Ferroso-ferric Phosphate. Composition perhaps

$$Fe_3(PO_4)_2.FePO_4.12H_2O.]$$

Ferrous sulphate	30 parts
Sodium phosphate	28 parts
Sodium acetate	10 parts

Dissolve the ferrous sulphate and the sodium phosphate each in 300 parts of boiling water, filter, and allow the solutions to cool to about 40° to 50° C. (104° to 122° F.). Dissolve the sodium acetate in 50 parts of warm water. Add the solution of ferrous sulphate to the solution of sodium phosphate, and lastly add the solution of sodium acetate. Mix well. As soon as the precipitate has subsided, decant the mother liquor. Wash the precipitate, either by decantation or on a muslin strainer, with hot water, until the washings cease to give a precipitate with solution of barium chloride. Dry the product at not over 50° C. (122° F.).

Reaction.

$$3FeSO_4.+2Na_2PHO_4=Fe_3(PO_4)_2+2Na_2SO_4+H_2SO_4.$$

The sulphuric acid then reacts with the sodium acetate, forming free acetic acid, in which the phosphate of iron is but sparingly soluble, and the precipitated ferrous phosphate is partially oxidized.

Notes. Unless sodium bicarbonate, sodium acetate, or sodium phosphate in excess be added, a portion of the ferroso-ferric phosphate remains in solution in the free acid formed by the reaction, and loss ensues. By neutralizing the free sulphuric acid with sodium bicarbonate, which does not affect the precipitate already formed, all of the phosphate of iron is thrown down. When first formed the precipitate is grayish-white or bluish-gray; it soon becomes grayish-blue, and should retain this color until washed and dried. The color may be easily spoiled, however, by too high heat, or by too long exposure to the action of the mother liquor, or to the air during the process of washing and drying. Instead of bluish the color may become greenish-gray. The surest way to obtain a handsome grayish-blue product is to employ sodium acetate, as directed in the foregoing formula. After drying, the product must be powdered and sifted. Dried at a high heat, the preparation forms very hard lumps; but if dried by moderate heat it may be easily rubbed through a fine sieve without using much force.

This product is the Ferri Phosphas of the British Pharmacopœia and of the U. S. P. of 1870.

It should contain at least 47 per cent of ferrous phosphate. It may also contain some ferric oxide.

Description.—A fine, grayish-blue, odorless, tasteless, insoluble powder.

IRON PHOSPHATE SYRUP.

SYRUPUS FERRI PHOSPHATIS.

Precipitated ferrous sulphate	28 Gm
Sodium phosphate	25 Gm
Sodium bicarbonate	7 Gm
Phosphoric acid (50%)	136 Gm
Sugar	450 Gm

Dissolve the ferrous sulphate in about 300 ml of boiling water, and the sodium phosphate in a similar quantity of cold water; mix the solutions; add the sodium carbonate previously dissolved in 100 ml of water, and, after stirring well, transfer the precipitate to a muslin filter and wash it with distilled water until the filtrate ceases to be affected by test solution of barium chloride. Mix the washed and drained precipitate in a mortar with the phosphoric acid. As soon as the phosphate is dissolved, filter the solution into a bottle, add 400 ml of distilled water and the sugar, and shake until dissolved, without the aid of heat. Lastly, add sufficient distilled water to make the final product measure 660 ml.

Notes. The product contains about 1 Gm of anhydrous phosphate of iron ($Fe_2(PO_4)_2$) in one imperial fluidrachm. If the quantity obtained by the above formula be diluted with 30 ml of simple syrup, the preparation will then contain 1 Gm to each 4 ml.

Another Method.

Iron wire, polished, cut	7 Gm
Phosphoric acid (50%)	120 Gm
Distilled water	40 ml
Simple syrup	600 ml

Put the iron wire, acid, and water into a flask, being careful that the metal is completely covered by the liquid; insert a loose plug of cotton in the neck of the flask, and set it aside for two or three days. When the iron is dissolved, filter the liquid, add the syrup, and lastly enough distilled water to make the final product measure 820 ml.

Note. This preparation is identical with that of the British Pharmacopœia.

Description.—A clear, nearly colorless, thick syrup of acid and ferruginous taste.

IRON (FERRIC) PHOSPHATE; PRECIPITATED.

FERRI PHOSPHAS PRAECIPITATUS.

$FePO_4.2H_2O=187.$

Solution of normal ferric sulphate.....	660 ml
Sodium phosphate	450 Gm
Sodium acetate	170 Gm

Dilute the solution of ferric sulphate with 4000 ml of water; add the sodium acetate, and dissolve. Dissolve the sodium phosphate in 4000 ml of water, and filter, if necessary. Pour the solution of ferric sulphate and sodium acetate gradually into that of the sodium phosphate, stirring well. Wash the precipitate with warm water until the washings are free from sulphate. Collect and dry the precipitate with the aid of gentle heat.

Reaction. $Fe_2(SO_4)_3+2(Na_2HPO_4.12H_2O)+2(NaC_2H_3O_2.3H_2O)+4H_2O=2FePO_4.2H_2O+3(Na_2SO_4.10H_2O)+2HC_2H_3O_2.$

Notes. It will be seen from the equation that the sodium acetate is added simply to prevent the formation of free sulphuric acid, which would occasion loss of ferric phosphate, which is soluble in that acid but insoluble in acetic acid.

The precipitate is white or cream-colored and very voluminous. On this account it is difficult to wash. When the quantity operated upon admits of it, the washing should be effected by mixing the precipitate with warm water in a tall vessel, allowing it to settle, drawing off the clear supernatant liquid by means of a siphon, and then transferring the magma to a wetted muslin strainer, where it may be left to drain thoroughly. This operation is to be repeated if necessary.

When the precipitate is allowed to remain long in contact with the mother liquor it gradually becomes more dense; also when the washing is too long continued. If large quantities are operated upon the washing consumes several days.

Description.—A white, or cream-colored, odorless, tasteless, insoluble powder. Soluble in dilute orthophosphoric acid, but insoluble in dilute metaphosphoric acid (difference from the pyrophosphate).

Iron (Ferric) Phosphate; Soluble.

FERRI PHOSPHAS SOLUBILIS.

Ferric citrate	10 parts
Sodium phosphate	11 parts
Distilled water	20 parts

Dissolve the ferric citrate in the water with the aid of water-bath heat. Add the sodium phosphate to the solution, and stir constantly until it shall have been dissolved. Evaporate the solution over the water-bath, at a temperature not exceeding 60° C., to the consistence of thick syrup, spread it on glass plates, and let it dry to form scales.

The product should be kept in dark amber-colored bottles, tightly closed.

Notes. The solution of the ferric citrate in water is brownish-red; this color is not immediately changed on the addition of the sodium phosphate, but when the reaction has taken place by which ferric phosphate and sodium citrate are formed the solution turns green. The sodium phosphate used must not be to any degree effloresced.

Description.—Thin, green, clear, transparent scales, odorless, of an acidulous, slightly saline taste. Permanent in dry air when excluded from light. Becomes darkened and brownish on exposure to light. It is readily, freely and perfectly soluble in water, but insoluble in alcohol. The water-solution has a slightly acid reaction.

Syrup of the Phosphates of Iron, Quinine, and Strychnine.

Soluble ferric phosphate	20	Gm
Quinine sulphate	30	Gm
Strychnine	0.2	Gm
Phosphoric acid	48	ml
Glycerin	100	ml
Water	50	ml
Syrup.		

Heat the soluble ferric phosphate with the water, in a porcelain capsule, until it is dissolved. Then add the phosphoric acid, the quinine sulphate, and the strychnine, and stir, until solution is effected. Filter the liquid into the glycerin, contained in a graduated bottle, add enough syrup to make up the volume to one liter and mix thoroughly. Lastly, strain, if necessary.

Description.—A clear, pale-green syrup; odorless; of bitter, acid taste. The preparation shows a bluish fluorescence.

Ammonio-Ferric Citro-Phosphate.

[Soluble Phosphate of Iron with Citrate of Ammonium.]

Add the washed precipitated ferric phosphate obtained by the formula given on p. — from 66 ml of solution of normal ferric sulphate and 45 Gm of sodium phosphate, to 60 ml of di-ammonium hydrogen citrate prepared by the formula given under the head of Ammonium Citrate. Heat the mixture by means of a water-bath for three hours, stirring occasionally, taking care not to allow the temperature to exceed 60° C. Evaporate the solution to the consistence of syrup, spread it on plates of glass, and dry it in scales.

Notes. The ferric phosphate is added in slight excess in order to perfectly saturate the solution with it.

If 60 ml of solution of di-sodium-hydrogen citrate be used instead of the citrate of ammonium, the product will be the Pharmacopœial soluble "phosphate of iron."

IRON (FERRIC) PYROPHOSPHATE; PRECIPITATED.

FERRI PYROPHOSPHAS PRAECIPITATUS.

$Fe_4(P_2O_7)_3 = 746.$

Solution of normal ferric sulphate.....	500 ml
Sodium pyrophosphate	320 Gm

Dilute the solution of ferric sulphate with 3500 ml of water, and dissolve the pyrophosphate of sodium in 4000 ml of hot water. Filter the liquids. Pour the solution of ferric sulphate slowly into that of the pyrophosphate, stirring well. Wash the precipitate with warm water on a wetted muslin strainer until the washings are free from sulphate. Dry the product by the aid of gentle heat.

Reaction. $2Fe_2(SO_4)_3 + 3Na_4P_2O_7 = Fe_4(P_2O_7)_3 + 6Na_2SO_4.$

Notes. The solutions must be cold when mixed, and the iron solution must be added very slowly to that of the sodium salt, with constant and brisk stirring. The precipitate is very light and bulky, and hence quite difficult to wash.

Description.—A white or cream-colored, odorless and tasteless powder. Insoluble in water. Soluble in dilute metaphosphoric acid but insoluble in dilute orthophosphoric acid. [Difference from the phosphate (orthophosphate).]

Iron (Ferric) Pyrophosphate; Soluble.

FERRI PYROPHOSPHAS SOLUBILIS.

[Sodio-Ferric Citro-Pyrophosphate.]

Ferric citrate	5 parts
Sodium pyrophosphate	5 parts

Dissolve the citrate in 10 parts of distilled water heated on a water-bath, add the pyrophosphate, and continue the heating with constant stirring until the pyrophosphate of sodium is dissolved and the solution turns green. Evaporate at a temperature not exceeding 60° C. (140° F.) until the solution has the consistence of thick syrup; spread it on glass plates, and dry it in scales.

Notes. Sodium citrate has been substituted for ammonium citrate for the preparation of pyrophosphate of iron by the Pharmacopœia, on the ground that the ammonio-ferric salt became insoluble through loss of ammonia, and because it either darkened or became opaque by age. The sodio-ferric pyrophosphate, however, is also liable to become impaired by age and exposure to light. The preparation is difficult to scale satisfactorily. In small quantities it may be best scaled in the cold. Pyrophosphate of iron should be carefully protected against light. It is best preserved in amber-colored bottles, tightly corked, and kept in a dry, cool, dark place.

Soluble ferric pyrophosphate with ammonium citrate may be prepared by adding the precipitated ferric pyrophosphate obtained from 500 ml of solution of normal ferric sulphate and 320 Gm of sodium pyrophosphate to 460 ml of the solution of di-ammonium hydrogen citrate and digesting at not over 60° C. for three hours, or until no more of the ferric pyrophosphate dissolves. The solution is then filtered, evaporated, and the preparation scaled.

Description.—Thin, transparent, green scales; odorless; taste a little acidulous, slightly saline. Freely soluble in water. Insoluble in alcohol.

Another Method.

[After the Swiss Pharmacopœia.]

Sodium pyrophosphate	75 parts
Solution of ferric chloride, U. S. P.	97 parts
Citric acid	26 parts
Ammonia water,	
Distilled water, of each sufficient.	

Dissolve the sodium pyrophosphate in 500 parts of distilled water.

Dilute the solution of ferric chloride with 800 parts of distilled water.

Pour the iron solution a little at a time into the solution of sodium pyrophosphate, stirring uninterruptedly.

Wash the precipitate thoroughly in the usual way.

Dissolve the citric acid in 50 parts of distilled water with enough ammonia water to render the solution slightly alkaline.

Add the moist, precipitated ferric pyrophosphate to the solution of ammonium citrate, stir well, and heat at not over 50° C. until solution is effected.

Filter the solution, evaporate it, and scale the product in the usual way.

Solution of Sodio-Ferric Pyrophosphate.

[Solution Leras.]

Ferric chloride	6 parts
Sodium pyrophosphate	17 parts
Distilled water, sufficient.	

Dissolve the ferric chloride in 500 parts of distilled water.

Dissolve the sodium pyrophosphate in 500 parts of distilled water.

Mix the solutions. Filter.

Description.—A pale yellowish, clear liquid of alkaline reaction. Gently heated with an equal volume of acetic acid it yields a white gelatinous precipitate which is blackened by hydrogen sulphide.

It contains about 0.114 to 0.119 per cent of Fe.

IRON SACCHARATE, OR SOLUBLE SACCHARATED IRON.

FERRI SACCHARATUM SOLUBILE.

[Saccharated Oxide of Iron. Iron Sugar.]

Solution of ferric chloride (U. S. P.)	230 parts
Sodium carbonate	260 parts
Solution of sodium hydroxide (15%)	50 parts
Sugar, pure, granulated,	
Distilled water, each, sufficient.	

Dilute the solution of ferric chloride with 1500 parts of distilled water.

Dissolve the sodium carbonate in 1500 parts of distilled water.

Pour the solution of sodium carbonate, a little at a time, into the diluted solution of ferric chloride, during uninterrupted stirring, taking care, until toward the close, to wait after each addition of the sodium carbonate solution until the precipitate formed shall

have redissolved before adding the next portion of the alkali carbonate.

When finally all of the sodium carbonate solution has been added, the precipitate is to be washed by the affusion and decantation of distilled water until the washings, when diluted with 5 volumes of water, no longer give any precipitate with silver nitrate test-solution but only rendered opalescent by it.

Transfer the magma to a cloth strainer and let it drain. Express as much of the water from the drained magma as practicable by moderate pressure.

Put the still wet solid mass into a porcelain dish and add 500 parts of sugar, together with the solution of sodium hydroxide and mix well.

Heat the mixture over a water-bath until a clear solution is formed.

Evaporate the liquid, by the water-bath heat, during constant stirring, to dryness, and powder the residue.

Add enough sugar to make the total weight of the product 1000 parts and reduce it to a medium fine powder.

Notes. The product is a water-soluble ferric sodio-saccharate.

145 parts of crystalline ferric chloride may be used instead of 230 parts of the official (U. S. P.) solution.

When the sodium carbonate solution is added to the solution of ferric chloride effervescence takes place from the CO_2 liberated and the precipitate at first formed, consisting of ferric hydroxide, redissolves in the solution of ferric chloride. But when more than one-half of the sodium carbonate solution has been added and much sodium chloride is accordingly contained in the liquid, the precipitate formed afterwards redissolves no longer.

Description.—A red-brown powder having a sweet and slightly ferruginous taste. One part of it dissolved in 20 parts of boiling water forms a perfectly clear red-brown solution having a feebly alkaline reaction. It contains from 2.8 to 3 per cent of Fe.

Another Method.

Solution of ferric chloride.....	230 parts
Distilled water, cold	170 parts
Sugar, granulated	100 parts

Make a solution.

Add to this solution, gradually and with constant stirring, 400 parts of solution of sodium hydroxide of 15 per cent strength.

Let the mixture stand a few hours (until it becomes clear).

Then add 5000 parts of boiling water.

The precipitate which is now formed is then washed by the affusion and decantation of distilled water until the washings have but a feebly alkaline reaction and begin to be colored.

Transfer the well-drained precipitate to a porcelain dish and add 900 parts of powdered sugar, mix well, and dry the mixture over a water-bath, stirring constantly. Add enough powdered sugar to the dry residue to make the total weight of the product 1000 parts.

Notes. It is recommended that the diluted ferric chloride solution and the sodium hydroxide solution shall both be *cold* when mixed. The *boiling* water afterwards added makes the precipitate denser and easier to wash. When about one-half of the alkali solution has been added a precipitate is formed, but this gradually redissolves as the remainder of the alkali is added and the liquid becomes clear on standing a few hours. The boiling water is then added.

Saccharated iron should make a clear solution in 5 parts of distilled water.

"Sirupus Ferri Oxydati"

of the German Pharmacopœia is a syrup made out of equal parts of saccharated iron, distilled water, and simple syrup. It contains or represents 1 per cent of its weight of Fe.

IRON SUBCARBONATE.

FERRI SUBCARBONAS.

Ferrous sulphate	8 parts
Sodium carbonate	9 parts

Dissolve the salts, each in 40 parts of boiling water, filter, and pour the solution of iron sulphate into that of the sodium carbonate, with constant stirring. Wash the precipitate with cold water, first by decantation and afterwards on a muslin strainer, until the washings cease to be precipitated by test solution of

barium chloride. Then drain, and dry the precipitate without heat.

Reaction. $FeSO_4+Na_2CO_3=FeCO_3+Na_2SO_4$. The ferrous carbonate oxidizes, as explained in the note.

Notes. To insure that the sodium carbonate is in excess over the ferrous sulphate, which is necessary to insure complete double decomposition, add the solution of the iron salt to the solution of sodium carbonate. The solutions should be hot in order that the ferrous carbonate formed may be heavy, and also to prevent the formation of soluble bicarbonate. When cold solutions are used a portion of the precipitate, which is at first nearly white, soon decomposes, losing a portion of its carbonic acid, which combines with another portion of ferrous carbonate and holds it in solution as bicarbonate. After a time this, too, decomposes, the carbonic acid being liberated and ferric hydroxide is formed by the absorption of oxygen from the air. When hot solutions are used the carbonic acid is rapidly expelled and the perfect precipitation of the iron compound not retarded. Owing to gradual oxidation the precipitate rapidly changes color in contact with the air, passing through various shades of gray, green, blue, olive, brown, and red. The gray, green, blue and olive are due to ferroso-ferric hydroxide, while the brown and red are caused by ferric hydroxide. Finally, the precipitate becomes almost completely ferric. Dried at high heat the preparation is more red, and less readily soluble in hydrochloric acid.

The ferric hydroxide generally rises to the surface of the mixed liquids, and during the washing of the precipitate the surface of the magma becomes covered with it, while the interior continues bluish-gray or green. The washing is best effected with boiling water, and on a muslin filter. The pores of the filter-paper become rapidly closed by yellowish-red ferric hydroxide.

Dried at ordinary temperatures the preparation is olive-brown with a reddish tint, retaining a small amount of ferrous carbonate, and is easily powdered. Dried with the aid of considerable heat the product is brown-red, and cakes together in hard lumps, which are not so readily powdered.

Description.—A red or brown-red, odorless, tasteless, insoluble powder.

IRON (FERROUS) SULPHATE.

FERRI SULPHAS.

$FeH_2SO_5.6H_2O = 278$.

Iron in the form of clean, bright wire, cut small	3 parts
Sulphuric acid	5 parts
Water, sufficient.	

Put 15 parts of water in a flask or dish and add the sulphuric acid very gradually, stirring constantly. Then add the iron. When effervescence has nearly ceased, heat the contents to boiling for about ten minutes. Filter while hot. Set aside to crystallize.

Collect the crystals and dry them on filter paper.

Evaporate the mother liquor to one-half its weight, again set aside to crystallize, and collect the second crop of crystals in the manner as the first.

Reaction. $Fe+H_2SO_4+7H_2O=FeH_2SO_5.6H_2O+H_2$.

Notes. The iron is used in excess of amount required by theory in order that no ferric salt may be formed. The hydrogen, which is liberated in the chemical reaction, has a disagreeable odor from volatile carbon, sulphur, and phosphorus compounds.

To prevent contamination with ferric salt, the filtrate may be acidulated with sulphuric acid before being set aside to cool; any ferric sulphate present will remain in the mother liquor.

The crystals should be dried as rapidly as practicable to avoid oxidation, which easily takes place when the salt is moist. To facilitate the drying the crystals may be hastily washed with a little alcohol, which evaporates rapidly.

Recrystallized Ferrous Sulphate.

Green vitriol	10 parts
Water	15 parts
Diluted sulphuric acid	1 part

Make a solution with the aid of heat. Boil it until a filtered sample is of a bluish-green color. Then filter while hot, and set the solution aside to crystallize in the usual way. Drain and dry.

the crystals as rapidly as possible and at once bottle the product in a dry container to be tightly closed.

Notes. The crystals must have a bluish-green and not a yellowish-green color, and they must be perfectly clear. The ferric sulphate and other ferric compounds contained in the green vitriol are removed partly by the boiling and filtration, and partly in the crystallization when the ferric sulphate present remains in the mother liquor.

Granulated Ferrous Sulphate.

Ferrous sulphate	100 Gm
Boiling distilled water	100 ml
Diluted sulphuric acid	5 ml
Alcohol	25 ml

Dissolve the ferrous sulphate in the water, add the acid, and filter the solution while hot. Evaporate at once in a tared porcelain dish over a sand-bath until the weight of the liquid is reduced to 150 Gm. Then cool it quickly, stirring uninterruptedly.

Transfer the liquid and crystals to a glass funnel loosely stopped with absorbent cotton, and, when the liquid has passed off, pour the alcohol over the mass of crystals in the funnel. When the alcohol has passed through, transfer the crystals to bibulous paper and dry them quickly at the ordinary temperature and transfer the dry product at once to well-stoppered bottles.

Precipitated Ferrous Sulphate.

Iron wire	2 parts
Sulphuric acid	3 parts
Distilled water	12 parts
Alcohol	6 par

Add the sulphuric acid gradually to the water, stirring well. Add the iron to the warm mixture. When effervescence has nearly ceased heat the mixture at the boiling point for about ten minutes. Filter while hot. Add a small quantity of sulphuric acid to the filtrate. Then pour the solution into the alcohol, stirring constantly.

Collect the precipitated crystalline ferrous sulphate on a filter, transfer it to bibulous paper, and dry it quickly at the ordinary

room temperature, stirring the crystals often and changing the paper once or twice to facilitate rapid drying.

When thoroughly dry, put the product in dry bottles to be well corked or stoppered.

Notes. Iron is used in excess and the acid is saturated with it. Free sulphuric acid is added to the solution because the salt crystallizes best from an acid solution.

Ferrous sulphate is insoluble in alcohol, but ferric sulphate is soluble. Hence any ferric sulphate present will remain in the alcoholic liquid.

The crystals wet with alcohol instead of water dry rapidly and are moreover surrounded by an atmosphere of alcohol vapor instead of air, so that the process of drying is accomplished with far less danger of oxidation.

Turbidated Ferrous Sulphate.

Ferrous sulphate	100 parts
Distilled water	100 parts
Diluted sulphuric acid	5 parts
Alcohol	25 parts

Dissolve the ferrous sulphate in the water, heated to the boiling point, add the acid, and filter the hot solution. Evaporate the filtrate in a tared porcelain dish over a sand-bath until the weight of the contents is reduced to 150 parts and then cool it quickly, stirring constantly. Transfer the wet salt mass to a glass funnel the throat of which is loosely closed with a plug of absorbent cotton, and when the product has well drained pour the alcohol upon it. When the alcohol has passed through it spread the crystalline salt upon bibulous paper, dry it quickly at the ordinary temperature, and transfer it at once to perfectly dry bottles and close these tightly.

The product is a pale bluish-green crystalline powder. Its official title (U. S. P., 1890) is "Granulated Ferrous Sulphate."

Description.—Ferrous sulphate forms large, clear, transparent, bluish-green crystals, or a crystalline granular salt; odorless; taste saline, styptic, ferruginous. Efflorescent in dry air. On exposure it becomes first whitish on the surface, then yellowish, brownish or reddish from ferric compounds. Soluble in 1.8 parts of water at 15°, and in 0.3 part of boiling water. Insoluble in alcohol.

IRON (FERROUS) SULPHATE. DRIED.

FERRI SULPHAS EXSICCATUS.

Approximately $FeH_2SO_5=170$.

Crystallized ferrous monometa-sulphate contains 6 molecules of water. When moderately heated it dissolves in its water of crystallization; between 33° C. and 90° C. the salt loses that water. At 280° C. partial decomposition of the sulphate is liable to take place.

In exsiccating ferrous sulphate, liquefaction of the salt, by solution in its water of crystallization, must be avoided if a light-colored product, unoxidized and free from hard particles, is to be obtained. The ferrous sulphate should be pure, and granulated, precipitated, or reduced to coarse powder. It may then be spread out in a thin layer on paper and be placed in a drying room where the temperature is about 40° C. The drying may also be effected by the heat of the sun until thoroughly effloresced, or converted into a white powder. Occasional stirring is required. Should the temperature exceed 50° C. the salt partly dissolves in its water of crystallization and the liquid penetrates the paper. Delf plates may be used instead of paper, or porcelain or iron dishes.

If the salt is allowed to become so hot as to dissolve in its water of crystallization, it will oxidize more rapidly, and becomes discolored. After having effloresced, however, it will no longer liquefy even at water-bath heat, or higher temperatures.

If water-bath heat is used in finishing the exsiccation, several days' heating will be necessary to expel the last portions of the sixth molecule of water; but an almost white product may readily be obtained, amounting to about 64 to 65 per cent of the crystallized salt, without prolonging the heating over water-bath beyond a few hours, the powder being well stirred during the process.

To get rid of the remainder of the water it is necessary to raise the heat to about 120° to 150° C., continuing the heat until the product ceases to lose weight. This prolonged exposure inevitably results in a dark-colored product.

To avoid decomposition and discoloration the Pharmacopœia no longer requires the product to be absolute FeH_2SO_5, but allows some moisture in it.

It should be triturated to fine powder and sifted.

Dried ferrous sulphate must be put in dry bottles.

One Gm of dried ferrous sulphate corresponds to about 1½ Gm of the crystallized salt.

Description.—A fine, grayish (almost white) powder, slowly but completely soluble in water.

Iron (Ferrous) Ammonium Sulphate.

Ferrous sulphate	100 parts
Ammonia water	123 parts
Diluted sulphuric acid	360 parts

Add the diluted sulphuric acid gradually to the ammonia water, stirring well. Dissolve the ferrous sulphate in the mixture. Filter, if necessary. Evaporate the solution to crystallization.

Description.—The product is not a double salt but a mixture of an equal number of molecules of ferrous sulphate and ammonium sulphate. It is pale-green, readily water-soluble.

IRON SUBSULPHATE.

FERRI SUBSULPHAS.

(*Basic Ferric Sulphate. Monsel's Powder.*)

When solution of basic ferric sulphate is evaporated at a moderate heat upon a delf or glass plate, the residue consists of transparent scales, which deliquesce on exposure. When dried at a higher temperature, the salt turns yellow and becomes less hygroscopic. When the solution is painted upon an iron plate with a brush, and the plate heated over a naked flame, the residue is a porous crust, which should be at once removed, and powdered while still hot in a warm mortar. The product thus obtained is yellow, quite hygroscopic, and perfectly uniform.

If the solution is evaporated to dryness in a dish, stirring the drying mass, and continuing the heat until a dry powder is formed, the product is a coarse, grayish-yellow, gritty powder, not as readily soluble in water.

Monsel's powder should be a fine, yellow, hygroscopic powder. It must be kept in a tightly corked bottle.

Solution of Basic Ferric Sulphate.

(Monsel's Solution.)

An aqueous solution containing about 43.5 per cent of basic ferric sulphate of varying chemical composition, consisting perhaps principally of a compound represented by the formula $Fe_4O(SO_4)_5$=720. The proportion of iron compound in the solution corresponds to about 13.6 per cent of metallic iron. It is prepared as follows:

Ferrous sulphate.....	135 parts
Sulphuric acid, 92.5%	14 parts
Nitric acid, 68%.	
Distilled water, each, sufficient.	

Add the sulphuric acid gradually to 100 parts of distilled water in a roomy porcelain dish, pouring the acid slowly and in a thin stream into the water, stirring constantly with a glass rod. Heat the mixture to a temperature of nearly 100° C.; add 15 parts of nitric acid and mix well.

Divide the coarsely powdered ferrous sulphate into four equal portions, and add these portions, one at a time, to the hot liquid, waiting after each addition until effervescence has nearly ceased.

When all the ferrous sulphate has been added, and has dissolved, add a few drops of nitric acid, and, if this causes a further evolution of red fumes continue to add more nitric acid, a few drops at a time, until it no longer causes nitrous vapors to be evolved.

Then boil the solution until it assumes a ruby-red color and is free from nitrous odor.

Lastly add enough distilled water to make the total product weigh 200 parts.

Filter if necessary.

Keep the product in glass-stoppered bottles in a moderately warm place (not below 22° C.), protected from light.

Reaction.

$$12FeH_2SO_5.6H_2O+3H_2SO_4+4HNO_3$$
$$=3(Fe_4O(SO_4)_5)+12H_2O+4NO.$$

Notes. See the notes under the title of Solution of Ferric

Sulphate. Note that the only difference between the formulas for the preparation of solution of normal ferric sulphate and the solution of basic ferric sulphate, respectively, is that about twice as much sulphuric acid is employed in proportion to the ferrous sulphate to make the normal ferric sulphate as is prescribed for making the subsulphate.

The quantity of nitric acid prescribed is a little scant.

Solution of basic ferric sulphate sometimes crystallizes and forms a semi-solid whitish mass, if kept in a room too cold. Should this happen, the mass may be easily liquefied again by warming it. If kept in a sufficiently warm place, as directed, it does not crystallize.

This solution is also called "solution of subsulphate of iron."

Description.—A clear dark red-brown liquid; almost odorless; taste strongly styptic, acid. Sp. w. about 1.550 at 15°.

On slowly mixing 2 volumes of the solution with 1 volume of concentrated sulphuric acid, in a beaker, a semi-solid white mass will separate on standing (difference from *tersulphate*).

IRON (FERRIC) SULPHATE SOLUTION.

LIQUOR FERRI TERSULPHATIS.

(Solution of Tersulphate of Iron.)

An aqueous solution of normal ferric sulphate
$[Fe_2(SO_4)_3=400]$
containing about 28.7 per cent of that salt, corresponding to about 8 per cent of metallic iron.

Ferrous sulphate	400 Gm
Sulphuric acid (92.5%)	77 Gm
Nitric acid (68%).	
Distilled water, each, sufficient.	

Add the sulphuric acid gradually and with constant stirring to 200 milliliters of distilled water in a capacious porcelain dish; heat the mixture to 100° C.; then add 44 Gm of nitric acid, and mix well.

Divide the ferrous sulphate, coarsely powdered, into four equal portions, and add these portions, one at a time, to the hot liquid,

waiting after each addition until effervescence has nearly ceased.

When all the ferrous sulphate has been added and has dissolved, add a few drops of nitric acid, and, if this causes a further evolution of red fumes, continue to add more nitric acid, a few drops at a time, until it no longer causes red fumes to be evolved.

Then boil the solution until it assumes a reddish-brown color and is free from nitrous odor.

Lastly, add enough distilled water to make the whole product weigh 1,000 Gm. Filter, if necessary.

Reaction. $6FeH_2SO_5.6H_2O+3H_2SO_4+2HNO_3$

$=3Fe_2(SO_4)_3+11H_2O+2NO.$

Notes. The ferrous sulphate used must be in clear crystals and dry; or, in other words, it must be dry but not in any degree effloresced. If granulated, precipitated or turbidated ferrous sulphate is used it need not be powdered at all.

The oxidation of ferrous sulphate to ferric sulphate by nitric acid takes place much below 100° C. (212° F.). Water-bath heat is, therefore, sufficient for this purpose. The process is, however, hastened by the application of a somewhat higher degree of heat.

To oxidize all the ferrous salt is not difficult, a sufficient amount of nitric acid being used. But the preparation must be free from nitric acid when finished, and it is difficult to expel the excess which is almost always added in order to complete the oxidation. Hence, great care shou[1] be taken not to use more nitric acid than is necessary.

But the presence of a trace of nitric acid is less objectionable in this preparation than the presence of ferrous salt.

When the ferrous salt is being raised to ferric by means of the nitric and sulphuric acids, a very dark-brown or almost black compound is formed by the ferric salt with the nitrogen oxide. Hence the liquid grows nearly black at times and the evolution of nitrous vapors is at the same time temporarily arrested. But when the heat is then increased a sudden and copious evolution of "red fumes" results when the dark colored compound is decomposed, and the liquid at once becomes lighter in color. Should more ferrous sulphate be added before that decomposition has taken place and while the solution is still dark, or should the

ferrous sulphate be added all at once, or too rapidly, the effervescence, suspended for a while, may suddenly take place with such violence as to cause the liquid to boil over. Hence the liquid should be heated after each addition of ferrous sulphate until the dark color disappears before any more ferrous sulphate is added, and finally the product must be heated until reddish-brown instead of brownish-black and until free from nitrous odor.

When a few drops of the solution is added to a little freshly prepared test-solution of potassium-ferricyanide there should be no blue coloration or precipitate but at most only a greenish-brown coloration. This test proves the absence of ferrous salt.

The solution of ferric sulphate (U. S. P.), when completely precipitated with an excess of ammonia water (10%), yields about 14.84 per cent of ferric hydroxide. Compared with solution of ferric chloride (U. S. P.), 100 parts of solution of ferric sulphate contains the same amount of iron as 61.73 parts of solution of ferric chloride.

Description.—A clear, dark, red-brown liquid, almost odorless (having at most a very faint odor from traces of free nitric acid); taste strongly styptic, acid. Reaction acid. Sp. w. about 1.320 at 15°.

On slowly mixing 2 volumes of the solution with 1 volume of concentrated sulphuric acid, in a beaker, no solid white mass will separate on standing (difference from *subsulphate*).

The "liquor ferri persulphatis" or "solution of ferric sulphate" of the British Pharmacopœia is stronger than the American preparation. It has the sp. w. 1.441 and contains about 36.19 per cent of anhydrous ferric sulphate, corresponding to about 10.09 per cent of metallic iron. Thus 1,000 Gm of the American "solution of ferric sulphate" exactly equals 793 Gm of the British "solution of ferric sulphate."

IRON ALUM.

FERRI ET AMMONII SULPHAS.

[Sulphate of Iron and Ammonium. Iron Alum.]

$FeH_4N(SO_4)2.12H_2O=482.$

Solution of normal ferric sulphate.....	300 ml
Ammonium sulphate.....	40 Gm
Diluted sulphuric acid.....	30 ml

Heat the iron solution to the boiling point; add the ammonium sulphate, and, when this has dissolved, add the acid. Stir well. Set the mixture aside in a well covered vessel, in a cool place, for a day or two. Remove the crystals from the mother liquor, wash them hastily with a little cold water, dry them at once by pressing them gently between blotting paper, and put them, without delay, in a bottle, which must be tightly stoppered and kept in a cool place.

The crystals should be clean, well developed, and of a handsome violet color.

Reaction.

$$Fe_2(SO_4)_3+(H_4N)_2SO_4+24H_2O=2FeH_4N(SO_4)_2.12H_2O.$$

Notes. The sulphuric acid is added, because good crystals are best obtained when a slight excess of acid is present. If there is a deficiency of acid the crystals will be brownish, unclear, or very pale.

The color of the crystals of iron alum varies materially; it is sometimes bluish or purplish, sometimes violet, and sometimes a pale rose color. Crystals which are purplish violet may become almost devoid of color upon recrystallization from a water-solution unless the solution is rendered decidedly acid by the addition of H_2SO_4.

[Crystals of common alum grow in a saturated solution of iron alum. Large, clear, well defined crystals can thus be made which have a colorless center enveloped in a beautifully colored exterior. This is an interesting experiment for students to make.]

Description.—A violet colored salt, resembling alum in crystalline form, insoluble in alcohol, readily soluble in water, giving a brown solution.

Recrystallized Iron Alum.

Iron alum, when effloresced, discolored, or otherwise unsightly, may be recrystallized as follows:

Iron alum	100 parts
Water	300 parts
Diluted sulphuric acid	10 parts

Dissolve the salt in the acidified water with the aid of heat, filter the solution, and crystallize in the usual way.

IRON (FERROUS) SULPHIDE.

FERRI SULPHIDUM.

$FeS=88$.

Metallic iron in the form of wire, filings, turnings or scraps	7 parts
Sulphur	4 parts

Heat together in an earthenware crucible until fused.

Reaction. $Fe+S=FeS$.

Description.—Heavy, black, lustreless, odorless pieces or masses, readily soluble in hydrochloric acid with copious evolution of hydrogen sulphide.

IRON TANNATE.

FERRI TANNAS.

Solution of ferric acetate	10 parts
Tannic acid	10 parts
Distilled water, sufficient.	

Mix the solution of acetate of iron with 35 parts of distilled water, and dissolve the tannin in 75 parts of distilled water. Filter the liquids.

Add the tannin solution slowly to the iron solution, stirring constantly.

Wash the precipitate and dry it.

Description.—An odorless and tasteless, insoluble black powder.

IRON (FERRIC) VALERATE.

FERRI VALERIANAS.

$Fe(C_5H_9O_2)_3=359$.

Sodium valerate	5 parts
Solution of ferric chloride, U. S.	6 parts
Distilled water.	

Dissolve the sodium valerate in 60 parts of distilled water and

filter. Mix the solution of ferric chloride with 100 parts of distilled water. Add the iron solution gradually to the solution of sodium valerate, stirring well, taking care to discontinue the addition of ferric chloride as soon as it no longer causes precipitation. Collect the precipitate on a filter and wash it quickly with cold distilled water until the washings are tasteless. Dry the product on blotting paper frequently changed and without the aid of heat.

Keep the product in small, tightly closed bottles in a cool, dark place.

Reaction.

$$FeCl_3+3NaC_5H_9O_2=Fe(C_5H_9O_2)_3+3NaCl.$$

Description.—A brick-red, or reddish brown powder having an odor of valeric acid. Decomposed by boiling water. Soluble in alcohol.

LEAD ACETATE.

PLUMBI ACETAS.

$$Pb(C_2H_3O_2)_2.3H_2O=378.5.$$

(*Sugar of Lead.*)

Litharge	5 parts
Acetic acid	8 parts
Water	4 parts

Mix the litharge, acid and water in a porcelain dish. Stir well. Let stand a day. Then heat over a water-bath until the oxide is dissolved. Filter. Evaporate until a pellicle forms, and then set aside to cool and crystallize, adding first, if necessary, a little more acetic acid to impart to the solution a decidedly acid reaction. Collect the crystals, drain and dry them, immediately put the product in dry bottles, close these tightly, and keep them in a cool place.

Reaction.

$$PbO+2HC_2H_3O_2+2H_2O=Pb(C_2H_3O_2)_2.3H_2O.$$

Notes. The acetic acid dissolves the lead oxide without the aid of heat if sufficient time is allowed. But the solution is

greatly hastened by the application of moderate heat. Too high a temperature would cause a loss of acetic acid.

Should the solution have a green color this shows the presence of copper which is sometimes contained in litharge. To remove this copper place a strip of pure lead in the solution; the copper is then deposited upon the lead and may be scraped off after which the bright clean lead is again placed in the solution. This must be repeated as long as any copper deposits upon the lead. When the liquid is free from copper, add a little more acetic acid, *if necessary,* to render the solution decidedly acid in its reaction on test-paper.

When too concentrated, solutions of lead acetate can not easily be filtered through paper. Should the solution be found too concentrated (by evaporation) to pass through the paper filter readily, dilute it with distilled water.

Should the crystals obtained contain iron acetate they will have a yellowish or rusty color. The salt must then be redissolved and recrystallized until colorless.

When perfectly clear and colorless crystals are formed the product is pure.

The mother liquor may be evaporated to one-half its volume, and, after adding a little more acetic acid, set aside for the formation of an additional crop of crystals.

Very large crystals can be obtained by the spontaneous evaporation of a solution saturated at the temperature of the laboratory; but very large crystals are not desirable for pharmaceutical uses. Smaller needle-shaped crystals are to be preferred, and may be obtained on cooling solutions saturated at an elevated temperature. Very small crystals are obtained by turbidation; but lead acetate in very small crystals is too liable to become contaminated with carbonate on exposure to the air.

This salt must be drained and dried as rapidly as possible, but without the aid of heat, or at a very moderate temperature for the crystals are liable to dissolve in adhering mother liquor if wet, or to effloresce if dry.

When large amounts are prepared the crystals may well be drained on a muslin strainer and dried in an atmosphere of acetic acid produced by sprinkling a little of the acid about the drying salt. The product, if the quantity is not too large, may be most conveniently and safely dried by gently pressing it between blot-

ting paper, changing the paper several times and as soon as moist, until the salt is dry.

Moist lead acetate rapidly absorbs carbonic acid from the air, if exposed, and forms insoluble carbonate. The surface of the crystals becomes white when covered with carbonate.

The salt also easily loses water of crystallization.

Lead acetate must therefore be quite dry before it is bottled, the containers must be dry, quite filled, tightly closed, and put in a cool place.

Purification.

Commercial "sugar of lead" may be purified by re-crystallization.

Dissolve 60 parts of sugar of lead in 80 parts of hot distilled water, add 1 part of acetic acid, filter the solution, let it cool, and, when cold, collect the crystals. Evaporate the mother-liquor to obtain further crops of crystals, and continue this as long as a pure product is obtained.

Collect and dry the product as indicated in the "notes" above.

Description.—Colorless, clear, crystals of a faintly acetous odor, and sweetish, astringent, finally nauseous metallic taste. Efflorescent. Becomes whitish on exposure to air both from loss of water of crystallization and by the formation of carbonate due to the absorption of carbon dioxide from the air.

Soluble in 2.3 parts of water, and in 21 parts of alcohol, at 15°; in 1.5 parts of boiling water and in 1 part of boiling alcohol.

At 40° it loses its water of crystallization, which amounts to 14.25 per cent; but it loses a portion of its water even at 15° on exposure.

A solution of 10 per cent strength, if made with distilled water which has just been boiled to expel air and carbon dioxide, must be almost if not quite clear.

Recrystallized Lead Acetate.

Sugar of lead	100 parts
Water	200 parts
Acetic acid	5 parts

Dissolve the sugar of lead in the water by the aid of heat, add the acetic acid, mix well, filter, and set the solution aside to

crystallize at rest. Collect and drain the crystals, press them gently between cloths or bibulous paper, dry rapidly in cold air, and keep the product in tightly closed bottles.

Turbidated Lead Acetate.

Sugar of lead	30 parts
Boiling distilled water	30 parts
Acetic acid	1 part

Dissolve, filter, evaporate over a water-bath to one-half. Cool the solution quickly, stirring frequently. Collect, drain, and dry the crystals at the ordinary room temperature as quickly as possible with the aid of bibulous white paper, frequently changed. Put the product at once in dry bottles, close these tightly, and keep them in a cool place.

LEAD CARBONATE.

PLUMBI CARBONAS.

$2PbCO_3.Pb(OH)_2 = 773.5$.

Prepared by acting on sheet lead with acetic acid, moisture and carbonic acid gas, until the lead is consumed or covered with a thick coat of *white lead,* which is then removed, ground with water, washed and dried. Or, carbonic oxide is conducted through solution of subacetate of lead, the precipitate being the product, while the remaining liquor containing normal lead acetate is again converted into solution of subacetate of lead by maceration with levigated litharge.

White lead is a basic carbonate, as may be seen from the formula given above.

The same lead carbonate is also obtained when lead nitrate or normal lead acetate is decomposed by sodium carbonate:

Lead nitrate	10 parts
Sodium carbonate	9 parts

Dissolve the lead nitrate and the sodium carbonate each in 100 parts of water, and filter the solutions separately. Pour the lead salt solution into the sodium carbonate solution, stirring constantly. Wash the precipitate with hot water; collect and dry it.

Reaction.

$$3Pb(NO_3)_2+3Na_2CO_3+H_2O$$
$$=2PbCO_3.Pb(OH)_2+6NaNO_3+CO_2.$$

Description.—A heavy, white powder, insoluble in water, but soluble with effervescence in acetic or nitric acid. Odorless and tasteless.

LEAD CHROMATE.

PLUMBI CHROMAS.

$PbCrO_4=322.5.$

(*Chrome Yellow.*)

Lead nitrate	25 parts
Potassium dichromate	11 parts
Water.	

Dissolve the lead salt in 35 parts of hot water, filter, and dilute the solution with 30 parts of cold water.

Dissolve the dichromate in 35 parts of hot water, filter, and dilute this filtrate with 65 parts of cold water.

Add the dichromate solution gradually to the lead nitrate solution, stirring well.

Wash the precipitate by affusion and decantation of water several times until the washings are free from nitrate. Collect the lead chromate on a muslin strainer, let it drain, press out the most of the remainder of the water with the aid of a screw press, break the press cake into pieces and dry it upon glass plates with the aid of moderate heat.

Reaction.

$$2Pb(NO_3)_2+K_2Cr_2O_7+H_2O$$
$$=2PbCrO_4+2KNO_3+2HNO_3.$$

Notes. There should be as nearly complete decomposition of both factors as possible. Hence it is advisable not to add all of the dichromate solution to the whole quantity of the lead salt at once. Reserve, instead, a small portion of the solution of lead nitrate;

add the dichromate solution gradually to the remainder of the lead nitrate until the dichromate is *slightly* in excess, which may be ascertained by the color of a filtered portion of the mixture, for the liquid, on filtration, remains colorless until an excess of dichromate has been added, after which it becomes yellow. When a filtered test-portion is found to be yellow, add cautiously enough of the reserved portion of lead nitrate solution to render the liquid colorless again.

Twenty-five parts of lead acetate and 20 parts of potassium dichromate may be used instead of 25 parts of lead nitrate and 11 parts of dichromate.

When "chrome yellow" is boiled with a dilute solution of sodium hydroxide it is converted into "chrome red," which is a basic lead chromate: $PbCO_4.PbO$.

Description.—Chrome yellow is an orange yellow insoluble pigment, in lumps or in powder.

LEAD IODIDE.

PLUMBI IODIDUM.

$PbI_2=459.5$.

Lead acetate	9 parts
Potassium iodide	8 parts
Acetic acid	1 part

Dissolve the acetate in 90 parts of cold water, add the acid, and filter. Dissolve the iodide in 80 parts of cold water and filter. Pour the lead salt solution into the solution of potassium iodide, stirring constantly. Collect the precipitate on a filter and wash it with cold water. Dry it between bibulous paper.

Reaction. $Pb(C_2H_3O_2)_2+2KI=PbI_2+2KC_2H_3O_2$.

Notes. The addition of acetic acid to the solution of lead acetate is to prevent the precipitation of white carbonate and yellowish white oxyiodide of lead. Lead iodide is not more soluble in acetic acid than it is in water.

Cold solutions are used not only because lead iodide is soluble to a considerable extent in hot water, but also to obtain a more

finely divided product. When hot liquids are used a portion of the iodide is held in solution until the liquid is cold, separating gradually in crystalline scales.

Lead iodide is also soluble in cold water, and thus there is always a slight loss, which is increased by washing. Hence the quantity of water used for washing the precipitate should be limited, and the washing should be performed on a filter instead of by decantation.

The lead acetate solution must be added to the solution of the iodide, for it is necessary to have the latter present in excess until the liquids are mixed. Should the order of mixing be reversed, or should the lead acetate be in excess, oxyiodide of lead is formed.

On the other hand, if the potassium iodide is in too great excess after all the lead acetate has been added, loss will be occasioned by the formation of soluble double salts of the iodides of lead and potassium.

Potassium acetate also dissolves lead iodide.

To obtain a larger yield, lead nitrate is used instead of acetate, the lead iodide being almost insoluble in a solution of potassium nitrate. The formula is then as follows:

Second Method.

Lead nitrate	1 part
Potassium iodide	1 part
Water, sufficient.	

Dissolve the lead nitrate in 8 parts of cold water, and the potassium iodide in 3 parts of cold water. Filter the solutions. Add the lead solution to the solution of potassium iodide, with stirring. Decant the mother liquor and throw it away. Collect the precipitate on a filter and wash it with cold water. Dry it between bibulous paper.

Crystallization. If a crystalline product is desired, dissolve the washed and still wet precipitate by boiling it with two hundred times its weight of water, filter, and let the solution cool very slowly. The lead iodide obtained from 10 Gm of lead nitrate would require nearly three liters of boiling water for its solution. To obtain a small quantity of crystallized lead iodide as a specimen, boil the precipitate with 200 times its weight of water, filter

while hot, and set the filtrate aside to cool slowly. The undissolved portion should be reserved separately.

Reaction. $Pb(NO_3)_2+2KI=2KNO_3+PbI_2$.

Description.—A heavy, bright-yellow powder; odorless and tasteless. Soluble in 2,000 parts of water at 15°, and in about 200 parts of boiling water.

LEAD NITRATE.

PLUMBI NITRAS.

$Pb(NO_3)_2=330.5$.

Lead oxide, in fine powder.....	23 parts
Nitric acid (68%).....	20 parts
Water	100 parts

Heat the oxide, acid and water together in a porcelain dish until the oxide has dissolved; filter the solution; acidify it by adding about 0.50 part of nitric acid; evaporate and crystallize.

Reaction. $PbO+2HNO_3=Pb(NO_3)_2+H_2O$.

Notes. If copper be present in the lead oxide the solution of lead nitrate will be bluish instead of colorless. Iron would make it yellowish. Copper may be removed by precipitation on metallic lead as described in the notes under plumbi acetas. Iron is removed by repeated recrystallizations.

Clear crystals cannot be obtained unless the solution of lead nitrate is decidedly acid; hence, after saturating the nitric acid with lead oxide, a small quantity of free nitric acid must be added to the solution before it is set aside to crystallize.

The solution must also be saturated only at the room temperature, and the crystals formed by spontaneous evaporation of the cold solution.

In the absence of free nitric acid the crystals obtained will be white or opaque; also when formed by cooling hot solutions.

The mother-liquor should be evaporated for more crystals.

Commercial impure lead nitrate may be purified by recrystallization from a water solution acidified with nitric acid, any copper or iron present being first removed as already described.

Description.—Colorless, transparent crystals, odorless, sweetish, astringent, taste finally metallic. Soluble in 2 parts of water at 15° and in 0.75 part of boiling water. Insoluble in alcohol. Reaction acid.

Crystallized Lead Nitrate.

Crude lead nitrate	10 parts
Water	20 parts
Diluted nitric acid	3 parts

Dissolve the lead nitrate in the water by the aid of heat, add the acid, filter the hot solution, and set it aside to crystallize. Collect, drain, and dry the crystals, and keep the product in a tightly closed bottle.

Notes. The crystals should be perfectly colorless and transparent. If unclear or whitish they should be redissolved and recrystallized from a water-solution acidified with nitric acid as before.

LEAD OLEATE.

PLUMBI OLEAS.

$Pb(C_{18}H_{33}O_2)_2$=768.5.

Lead acetate	100 Gm
White castile soap, in fine powder	165 Gm

Dissolve the lead acetate in ten liters of water; if decidedly milky, add to it, gradually, enough acetic acid to render the solution clear, being careful to avoid adding an excess of the acid. Dissolve the soap in 2,500 ml of hot water, and add this solution slowly to that of the lead acetate. Boil the mixture, reject the mother liquor, wash the precipitated oleate twice with boiling water, using ten liters each time. Separate the water from the product.

Reaction.

$$Pb(C_2H_3O_2)_2+2NaC_{18}H_{33}O_2 = Pb(C_{18}H_{33}O_2)_2+2NaC_2H_3O_2.$$

Notes. Prepared in this manner the lead oleate is a hard, brit-

tle, white plaster when cold. The yield is about 160 Gm. This lead oleate is more hard and brittle than lead plaster, which is made by boiling lead oxide with olive oil and water. It contains 28.95 per cent of lead oxide. Lead oleate obtained by dissolving lead oxide in oleic acid is usually too soft and sticky. An oleate of lead containing 20 per cent of lead oxide dissolved in an excess of oleic acid is readily obtained by digesting 4 parts of lead oxide in fine powder with 4 parts of oleic acid at about 60° C., stirring frequently until combined, the product being a soft yellowish ointment.

LEAD OXIDE.

PLUMBI OXIDUM.

PbO=222.5.

Lead oxide is prepared on a large scale by oxidizing the metal at high temperatures in special furnaces. The purity of the product formed depends upon the purity of the lead used, and the degree of oxidation on the temperature and the access of air. The color also depends upon the temperature at the time of oxidation, and further upon the subsequent process of cooling. When slowly cooled the lead oxide is yellowish-red; but when rapidly cooled it is reddish-yellow.

Massicot is a yellow powder usually containing a small amount of "red lead" or "minium." But very pure lead oxide of this kind can be obtained from some of the manufacturers of paints. Massicot is PbO.

Litharge is also PbO, but is obtained by the partial fusing together of the particles of the yellow massicot. Litharge is either yellowish-red or reddish-yellow, varying considerably in the shade of its color.

[*Red lead,* or *minium,* is not PbO, but Pb_3O_4 (or $2PbO.PbO_2$). This oxide is also obtained by the oxidation of the metal, or by carefully heating litharge in a plentiful supply of air.]

Lead oxide, PbO, may be made by heating lead nitrate or lead carbonate until completely decomposed. The residue is the oxide. When pure lead nitrate is employed a very pure lead oxide is obtained if the heat be continued until red vapors cease to be given off and the residue acquires a constant weight. The

nitrate should first be powdered, the heat gradually increased, and the powder stirred.

Should the residue be partly yellow and partly red, as it always will be if not thoroughly stirred or mixed, it is triturated until of perfectly uniform color.

Pure lead oxide is completely soluble in solution of normal acetate of lead.

LEAD PEROXIDE.

PLUMBI PEROXIDUM.

PbO_2=238.5.

Lead nitrate	5 parts
Chlorinated lime	8 parts
Water.	

Dissolve the lead salt in 30 parts of hot water. Triturate the chlorinated lime well with 60 parts of cold water and strain. Add the chlorinated lime solution in portions to the hot lead salt solution in a dish, stirring well. When about one-half of the solution of chlorinated lime has been added, heat the mixture until the light colored precipitate turns dark-brown. Then filter about 10 ml of the liquid and test it by adding a little of the chlorinated lime solution and warming.

If a precipitate is formed, add more chlorinated lime solution to the mixture in the dish. Test again from time to time in the same way until no further precipitation is caused by the chlorinated lime. Then heat the mixture nearly to the boiling point, stirring well. Let settle, and wash the precipitate with boiling water, by decantation, until the washings are tasteless or free from chloride or nitrate. Collect and dry the peroxide.

Reaction.

$$Pb(NO_3)_2+2Ca(ClO)_2+H_2O$$
$$=PbO_2+CaCl_2+Ca(NO_3)_2+2HCl+2O.$$

Description.—A heavy, dark-brown, insoluble powder.

LEAD TANNATE.

PLUMBI TANNATUM.

Lead acetate..... 8 parts
Tannic acid..... 9 parts
Distilled water, sufficient.

Dissolve the lead acetate and the tannic acid, each, separately, in ten times its weight of distilled water. Add enough of the tannin solution to the lead solution to precipitate the lead in the form of tannate, discontinuing the addition of the tannic acid whenever it ceases to produce further precipitation.

Wash the precipitate with distilled water until the washings no longer give an acid reaction on test-paper.

Dry the product with the aid of gentle heat.

Description.—A grayish-yellow, odorless, tasteless, insoluble powder.

The substantive noun "tannatum" is used instead of *tannas* to indicate that the composition is indefinite.

GLYCERITE OF TANNATE OF LEAD.

Oak bark, in coarse powder..... 175 Gm
Glycerin ..... 35 Gm
Solution of subacetate of lead, sufficient.
Water, sufficient.

Boil the bark with two liters of water for fifteen minutes. Strain. To the colature add gradually solution of subacetate of lead so long as a precipitate continues to be formed. Collect the precipitate on a wetted muslin strainer and wash it with water until the washings are tasteless. Let drain, and press the magma between bibulous paper until enough of the moisture has been removed from it to reduce its weight to 65 Gm. Mix this while still moist with the glycerin.

LEAD PLASTER.

EMPLASTRUM PLUMBI.

[Diachylon Plaster.]

Lead oxide..... 32 parts
Olive oil..... 60 parts
Water.

Mix the lead oxide, previously passed through a No. 80 sieve, intimately with about one-half of the olive oil, by trituration, and add the mixture to the remainder of the oil contained in a bright copper kettle of a capacity equal to at least four times the bulk of the ingredients. Then add 10 parts of boiling water, and boil the whole together, over a fire, constantly stirring with a wooden spatula, until a small portion, when dropped into cold water, is found to be pliable and tenacious. From time to time add a little water to replace that lost by evaporation. When the mixture in the kettle has acquired a whitish color and is perfectly homogeneous, transfer it to a vessel containing warm water, and as soon as the mass has sufficiently cooled, knead it well with the water so as to remove the glycerin, renewing the water from time to time, as long as it may be necessary. Finally divide the mass into rolls of suitable size.

Description.—A yellowish-white, tenacious plaster.

Another Method (*G. P.*)

Lead oxide	100 parts
Olive oil	100 parts
Lard	100 parts
Water.	

Mix the oil and lard by melting them together. Add the lead oxide previously sifted and then well mixed with 20 parts water. Boil the mixture, stirring well and uninterruptedly with a wooden spatula. Add from time to time about 3 parts of hot water to replace that lost by evaporation. Continue the boiling until the plaster is formed and all red color has disappeared.

See also directions in the method first described.

Notes. Lead plaster made with olive oil alone (without lard) is softer than that made with olive oil and lard, because the former plaster contains principally lead oleate with but little stearate, while the latter contains a greater proportion of stearate. The constituents of the lead plaster are oleate, palmitate, stearate and arachate of lead.

The lead oxide does not react readily with oil or fat except at a very high temperature and in the presence of water. In the absence of water a plaster can be made but only at so high a

temperature that some of the fixed oil is decomposed, and a dark plaster having a peculiar odor is then formed.

The heat must not be too high. If the plaster mass bubbles or spatters violently when water is added, this proves that the temperature is excessive. To avoid boiling over, or dangerous spattering and the burning of the product, the kettle should be taken from the fire (when the addition of hot water causes much commotion), the contents should then be diligently stirred, and a small amount of hot water added. The kettle may then be replaced. As long as the mass foams quietly and no irritant vapors arise there is enough water present; but when the hot mass ceases foaming more hot water must be added. Otherwise the plaster rapidly gets too hot and may be darkened.

Diligent and vigorous stirring with a broad wooden spatula is necessary throughout the whole process of emplastrification, and it should be effected in such a manner that the heavy lead oxide may be uniformly distributed through the mixture and not permitted to settle to the bottom of the kettle.

As long as the plaster mass retains any reddish tint there is still some lead oxide uncombined, and the boiling must be continued until that tint has given place to a whitish color.

Steam heat *under pressure* may be employed, but the process requires longer time. Direct fire under the kettle or dish is more satisfactory. But the process of boiling lead plaster always occupies several hours even with the employment of direct heat over a good fire.

After the glycerin has been washed out of the plaster by kneading it with water, the product should be heated sufficiently to expel the water mixed with it.

To improve its color it may be "pulled" while sufficiently plastic, afterwards rolled into cylindrical sticks on a plaster board kept moist with a slight amount of water, the hands of the operator being also kept moistened.

LEAD SUBACETATE SOLUTION.

LIQUOR PLUMBI SUBACETATIS; U. S.

(*Goulard's Extract.*)

A water solution containing about 50 per cent of basic lead acetate, having the composition $HOPbC_2H_3O_2$=282.5. [This is

equivalent to about 24.2 per cent of $Pb_2O(C_2H_3O_2)_2$, which is the composition assigned to the basic lead acetate contained in this solution by the definition given in the American Pharmacopœia.]

Lead acetate..... 17 parts
Lead oxide, in fine powder..... 10 parts
Distilled water.

Heat 70 parts of distilled water to boiling. Dissolve the lead acetate in the hot water. Transfer the solution to a bottle. Add the previously sifted lead oxide, in small portions, shaking well and frequently after each addition to prevent the oxide from caking together, and waiting after each addition until the portion added has been dissolved before adding another portion. When all of the lead oxide has been added and dissolved, add enough distilled water to make the total product weigh 100 parts, and filter the solution through paper in a well covered funnel.

Keep the product in well-stoppered bottles.

Reaction.

$$Pb(C_2H_3O_2).3H_2O+PbO=2HOPbC_2H_3O_2+2H_2O.$$

Notes. The lead acetate must be in good condition—i. e., in transparent crystals containing the full amount of water of crystallization and free from carbonate. The lead oxide also must be free from carbonate and from minium and other impurities. Unless these conditions are observed the solution can not have the required strength, for lead carbonate and minium are not soluble in a solution of lead acetate. For the same reason the distilled water should be boiled to expel carbon dioxide. After making the solution of lead acetate with the hot water, it is not necessary to longer maintain the high temperature. It is usually directed that the solution of acetate be boiled with the oxide; but the lead oxide dissolves as well even in a cold solution.

Several basic lead acetates are known, differing as to their solubility as well as their percentage of lead. All are prepared by dissolving lead oxide in a solution of lead acetate, the only difference being the proportion of oxide added. Different pharmacopœias, moreover, prescribe different proportions of water. The strength of the solution obtained can not be reliably determined

from the proportions of the materials, but will always be less than theory indicates, for there is always an undissolved white powder remaining, which consists of carbonate.

The molecular weights of lead acetate and lead oxide (378.5 and 222.5) are in the proportion of 17 to 10; 378.5 parts of lead acetate and 222.5 parts of lead oxide form 565 parts of $HOPbC_2H_3O_2$, which would make 2260 parts of solution of 25 per cent strength (that is, 25 per cent of $HOPbC_2H_3O_2$, having the molecular weight 282.5). The exact quantity of solution which should be obtained from 17 parts of lead acetate and 10 parts of lead oxide would be 102 parts. But only 100 parts of solution are directed to be prepared from 17 parts of acetate and 10 parts of oxide. With such approximately pure materials as may be obtained without any difficulty, a product containing fully 25 per cent of $HOPbC_2H_3O_2$ [or $Pb(C_2H_3O_2)_2.Pb(OH)_2$.] results from the formula given.

The proportions ordered by the British Pharmacopœia are 250 Gm of lead acetate and 175 Gm of lead oxide to make 1,000 ml of product (or 1,275 Gm).

The German Pharmacopœia orders 3 parts of lead acetate, 1 part of lead oxide, and 10 parts of water.

The Pharmacopœia of Norway prescribes 17 parts of lead acetate, 5 parts of lead oxide, and 78 parts of water.

Bottles containing solution of subacetate of lead soon become coated with lead carbonate, which forms a white crust on their sides. This white crust is most readily removed by nitric acid.

The preparation becomes whitish on dilution with water containing carbonic acid, sulphates, etc., and can be diluted so as to form a clear liquid only with distilled water.

Description.—A colorless solution with a sweetish taste and alkaline reaction. Sp. gr. about 1.237.

Glycerite of Subacetate of Lead.

GLYCERITUM PLUMBI SUBACETATIS.

Lead acetate	250 Gm
Lead oxide, in fine powder	175 Gm
Glycerin	1,000 Gm
Distilled water	600 ml

Mix in a porcelain dish and boil the mixture fifteen minutes. Filter and evaporate the filtrate to 1,000 ml.

Notes. This solution will bear dilution with distilled water, yielding a clear liquid. It has about the same strength as the official solution of subacetate of lead.

Diluted Solution of Lead Subacetate.

LIQUOR PLUMBI SUBACETATIS DILUTUS; U. S.

(*Aqua Saturni. Lead Water.*)

Mix 30 ml of solution of lead subacetate with 970 ml of distilled water, previously boiled and allowed to cool again.

LITHIUM BENZOATE.

LITHII BENZOAS.

$LiC_7H_5O_2=128.$

Lithium carbonate	3 parts
Distilled water	30 parts
Benzoic acid	10 parts

Put the lithium carbonate and water into a porcelain dish placed over a water-bath, warm gently, and add the benzoic acid gradually. When solution has been effected filter the liquid and evaporate the filtrate until a moist crystalline mass remains. Dry the mass at a temperature of about 30° C.

Reaction.

$$Li_2CO_3+2HC_7H_5O_2=2LiC_7H_5O_2+H_2O+CO_2.$$

Description.—A white crystalline salt, either odorless or of a faint benzoin odor, and cooling sweetish taste. Soluble in 4 parts of water at 15° C. and in 12 parts of alcohol; and in 2.5 parts boiling water.

LITHIUM BROMIDE.

LITHII BROMIDUM.

LiBr=87.

Prepared by four different methods:

(1) By neutralizing hydrobromic acid with lithium carbonate. (2) By double decomposition between lithium sulphate and potassium bromide, the potassium sulphate being separated by the addition of alcohol in which potassium sulphate is insoluble while the lithium bromide is soluble. (3) By treating a solution of ferrous bromide with lithium carbonate. (4) From lithium sulphate and barium bromide.

The solution is evaporated to dryness.

Description.—A white, granular salt; odorless; taste sharp, bitterish. Deliquescent. Soluble in 0.6 part of water at 15°, and in one-half that proportion of boiling water. Readily soluble also in alcohol and in ether. Neutral to litmus paper.

LITHIUM CARBONATE.

LITHII CARBONAS.

Li_2CO_3=74.

A white, odorless powder of alkaline taste and reaction. Soluble in 80 parts of water at 15°, and in 140 parts of boiling water.

Insoluble in alcohol.

[The solubility of lithium carbonate in cold and in boiling water is variously stated by different authorities.]

LITHIUM CHLORIDE.

LITHII CHLORIDUM.

LiCl=42.4.

Neutralize diluted hydrochloric acid with lithium carbonate, filter the solution, and evaporate to dryness, stirring constantly

toward the close of the evaporation so as to obtain a granulated product.

Description.—A white, granular powder; odorless; taste saline. Readily soluble in water and in alcohol. Neutral in reaction.

LITHIUM CITRATE.

LITHII CITRAS.

$Li_3C_6H_5O_7.4H_2O=282.$

Lithium carbonate	8 parts
Citric acid	15 parts
Distilled water	60 parts

Dissolve, filter, and evaporate to crystallization, or until a granular salt remains. Dry the product with the aid of gentle heat.

Keep it in tightly closed bottles.

Reaction. $3Li_2CO_3+2H_3C_6H_5O_7+5H_2O$

$=2Li_3C_6H_5O_7.4H_2O+3CO_2.$

Description.—A white crystalline or granular powder; odorless; taste faintly alkaline, cooling. Hygroscopic in moist air. Soluble in 2 parts of water at 15°, and in one-half its weight of boiling water. Insoluble in alcohol and in ether.

Effervescent Lithium Citrate.

Lithium carbonate	70 Gm
Sodium bicarbonate	280 Gm
Citric acid	370 Gm
Sugar, in fine powder, a sufficient quantity.	

Triturate the citric acid with about 200 Gm of sugar, and dry the mixture thoroughly. Then incorporate with it, by trituration, the lithium carbonate and sodium bicarbonate, and enough sugar to make the product weigh 1000 Gm.

It may be granulated in the same way as effervescent magnesium citrate.

Keep the powder in well-stoppered bottles.

Description.—A white, coarsely granular powder, readily soluble in water, with effervescence, forming an acidulous solution.

LITHIUM IODIDE.

LITHII IODIDUM.

$LiI = 133.5$.

Prepared by double decomposition between calcium iodide and lithium carbonate, which are heated together in water. $CaI_2 + Li_2CO_3 = 2LiI + CaCO_3$. It may also be made from iron, iodine and lithium carbonate, in a manner similar to the process for preparing lithium bromide from iron, bromine and lithium carbonate.

The solution is evaporated to dryness.

Description.—A white granular salt; odorless; taste acrid, bitterish; deliquescent. Freely soluble in water, alcohol, and ether.

LITHIUM PHOSPHATE.

LITHII PHOSPHAS.

$Li_2HPO_4 = 103$.

Lithium chloride	4 parts
Sodium phosphate	17 parts
Water.	

Dissolve the salts, each in 150 parts of water, filter the solutions, and pour the lithium chloride solution into the other, stirring well. Collect the precipitate on a filter and wash it with cold water.

Reaction. $Na_2HPO_4 + 2LiCl = Li_2HPO_4 + 2NaCl$.

Description.—An insoluble white powder, odorless and tasteless.

LITHIUM SALICYLATE.

LITHII SALICYLAS.

$LiC_7H_5O_3 = 144$.

Salicylic acid	75 parts
Lithium carbonate,	
Distilled water, of each sufficient.	

Mix the salicylic acid with 200 parts of distilled water in a porcelain dish and heat gently over a water-bath. Add the lithium carbonate gradually, stirring well, until the solution obtained is *nearly* neutralized (or but feebly, though distinctly, acid in reaction). Filter the solution, and evaporate it to dryness, stirring constantly.

Keep it in well-stoppered bottles.

Reaction. $Li_2CO_3+2HC_7H_5O_3=2LiC_7H_5O_3.H_2O+CO_2$.

Notes. The quantity of lithium carbonate required for 75 parts of salicylic acid is about 20 parts. The acid should not be completely neutralized with lithium carbonate; still less should the solution be made at all alkaline for the salt would then soon become discolored. The filter paper used must be white filter paper, entirely free from iron; otherwise the product will be reddish.

Description.—A white, odorless powder, having a sweetish, somewhat disagreeable taste. Deliquescent on exposure.

Readily soluble in water and in alcohol. Its water-solution should have a slightly acid reaction on litmus paper.

MAGNESIUM ACETATE.

MAGNESII ACETAS.

$Mg(C_2H_3O_2)_2=142.2$.

Magnesium carbonate	40 parts
Acetic acid (36%)	137 parts

Dissolve the carbonate in the acid, with the usual precautions to prevent overflowing on account of the effervescence. Heat the liquid to expel carbon dioxide. Filter. Evaporate to dryness.

Description.—A white, amorphous, gummy, deliquescent salt. Freely soluble in water and in alcohol.

Must be kept in tightly closed bottles.

Solution of Magnesium Acetate.

This solution is said to be an effective antiseptic:

Acetic acid (36%)	40 parts
Magnesium carbonate	12 parts
Distilled water, sufficient.	

Heat the acetic acid gently in a porcelain dish over a water-bath. Add gradually the magnesium carbonate previously mixed with 20 parts of distilled water, stirring well. When effervescence has ceased, test the solution on litmus paper. If found acid, add enough magnesium carbonate to exactly neutralize it. Filter, and evaporate the filtrate to 30 parts.

MAGNESIUM BOROCITRATE.

MAGNESII BOROCITRAS.

Magnesium oxide	3 parts
Boric acid, in powder	3 parts
Citric acid, in powder	10 parts
Distilled water	4 parts

Mix the powders intimately; add the water and work the mixture into a mass. Let the mass harden; then triturate it to powder.

MAGNESIUM CARBONATE.

MAGNESII CARBONAS.

$$4MgCO_3.Mg(OH)_2.aq.$$

Prepared from chloride or from sulphate of magnesium by precipitation with sodium carbonate. The magnesium salts are obtained from the "bittern" of salt-works. Magnesium carbonate is also manufactured from *magnesite,* and from *dolomite,* or magnesian limestone, by dissolving the magnesium carbonate from the powdered mineral by means of cold water and carbonic acid under pressure, when magnesium bicarbonate is formed. The solution is then boiled, when the basic carbonate, which constitutes the official preparation, is precipitated.

In pharmacy distinction is made between "heavy magnesium carbonate" and "light magnesium carbonate." The light magnesium carbonate differs from the heavy variety in these respects: the light carbonate is about three times as bulky; when examined under the microscope, it is found to be partly amorphous, but containing numerous slender prismatic crystals, whereas the heavy variety is wholly amorphous and somewhat granular; the light magnesium carbonate yields less magnesium oxide on calcination.

These differences depend upon the methods employed in the preparation of the products. The working formulas given in the British Pharmacopœia are as follows:

Light Magnesium Carbonate.

Magnesium sulphate	25 parts
Sodium carbonate	30 parts

Dissolve each salt in 200 parts of cold distilled water, filter, and mix the solutions *cold.* Boil the mixture in a porcelain dish for fifteen minutes. Transfer the precipitate to a wetted muslin strainer, and wash it with boiling distilled water until the washings cease to give a precipitate with test solution of barium chloride. Then let the precipitate drain, and dry it at not over 100° C.

Heavy Magnesium Carbonate.

Magnesium sulphate	25 parts
Sodium carbonate	30 parts

Dissolve the salts, each in 50 parts of boiling distilled water; filter, and mix the two solutions while *hot.* Evaporate the mixture on a sand-bath to perfect dryness. Digest the residue for half an hour with 100 parts of boiling water, collect the precipitate on a wetted muslin strainer, and wash it repeatedly with distilled water until the washings cease to give a precipitate with test solution of barium chloride. Drain the product and dry it at not over 100° C.

Reaction. $4MgSO_4+4Na_2CO_3+5H_2O=3MgCO_3.Mg(OH)_2.4H_2O+4Na_2SO_4+CO_2$; or perhaps $5MgSO_4+5Na_2CO_3+6H_2O=4MgCO_3.Mg(OH)_2.5H_2O+5Na_2SO_4+CO_2$.

Notes. It will be seen that the differences in manipulation are: the use of more dilute and cold solutions in making the light magnesium carbonate, and the precipitate is in that case simply heated for fifteen minutes with the mother liquor; while, in making the heavy magnesium carbonate, the solutions are four times as concentrated, and mixed while hot, the mixture being then

evaporated to perfect dryness on a sand-bath, after which the residue is freed from sodium sulphate by digestion and subsequent washing with boiling water.

When the precipitate is formed with cold solutions, it contains rather less magnesium than if prepared with hot solutions. When boiled in the liquid for a short time carbonic acid passes away; in preparing the light magnesium carbonate the heating should be discontinued as soon as the evolution of carbonic acid has commenced. When formed with hot, concentrated solutions, the mixture being boiled down to dryness, the precipitate contains less carbonic acid when formed, and loses more of that acid afterwards when exposed to the stronger heat.

Composition. Fritzsche, Hager and Flückiger state that the composition of magnesium carbonate is $4MgCO_3.Mg(OH)_2.4H_2O$; the U. S. P. gives it as $4MgCO_3.Mg(OH)_2.5H_2O$; the British Pharmacopœia states it as being $3MgCO_3.Mg(OH)_2.4H_2O$. That there is a difference in composition between light and heavy magnesium carbonate may be regarded as certain, although the same formula is given by the British Pharmacopœia for both. Light magnesium carbonate contains a greater proportion of true carbonate ($MgCO_3$) and a correspondingly less proportion of hydroxide ($Mg(OH)_2$). The quantity of water in the product seems also to vary.

Description.—Light, white, friable masses, or a light, white powder; odorless; produces a slightly earthy feel in the mouth. Practically insoluble in pure water, although it imparts to it a slightly alkaline reaction. Insoluble in alcohol.

The foregoing description applies to the light variety. The heavy carbonate is a powder only one-third as bulky.

MAGNESIUM CHLORIDE; CRUDE.

MAGNESII CHLORIDUM CRUDUM.

$MgCl_2.6H_2O$=203.

Magnesite, in powder	100 parts
Hydrochloric acid (32% of HCl)	260 parts
Magnesium oxide	1 part
Water	50 parts

Mix the magnesite with the water in a large porcelain dish; heat to about 90°, and add the acid in small portions at a time, stirring well and allowing the effervescence to subside after each addition before adding more. When all of the acid has been added heat the liquid to 90° for a few minutes. Neutralize perfectly with magnesia. Add 50 parts of water and mix well. Then add 5 parts of chlorine water, stir well, and keep the mixture hot for about one hour. Filter while hot. Evaporate the filtrate until it has about 1.30 sp. w. while still hot. Then allow it to cool. Collect and drain the crystals.

Reaction. $MgCO_3+2HCl=MgCl_2+H_2O+CO_2$.

Notes. The product contains some calcium chloride because the magnesite contains calcium carbonate. The solution of magnesium chloride should not be allowed to boil, as the salt would then be liable to decompose to some extent.

Description.—Colorless, deliquescent crystals, soluble in less than two-thirds of their own weight of cold water.

Pure Magnesium Chloride.

May be prepared by saturating pure hydrochloric acid with pure magnesium carbonate, filtering the solution and evaporating to crystallization.

MAGNESIUM CITRATE.

MAGNESII CITRAS.

$Mg_3(C_6H_5O_7)_2.aq.$

In the Swedish Pharmacopœia occurs a magnesium citrate of the formula indicated, but mixed with an excess of citric acid amounting to about 9 per cent. It is prepared as follows:

Magnesium carbonate, in powder	5 parts
Citric acid, in fine powder.....	8 parts
Alcohol	12 parts

Mix the magnesium carbonate and acid intimately by trituration in a mortar, and add the alcohol, mixing the whole well so

as to form a pasty mass free from lumps. Cover the mortar loosely with paper, and set it aside until the alcohol has evaporated, leaving a dry residue, which must be at once reduced to powder.

Keep the product in well stoppered bottles.

Reaction. $3(4MgCO_3.Mg(OH)_2)+10H_3C_6H_5O_7=$

$$5Mg_3(C_6H_5O_7)_2+12CO_2+18H_2O.$$

Notes. To insure as complete a chemical union as practicable the citric acid might be dissolved in the alcohol, and the magnesium carbonate added in small quantities at a time. On the addition of the magnesium carbonate the temperature of the mixture falls considerably. The mixture soon becomes a hard mass unless triturated without interruption until rendered perfectly homogeneous. The carbon dioxide escapes with effervescence, and the alcohol is added to facilitate the reaction and the liberation of the gas. To prevent too rapid dissipation of the alcohol, the mass is allowed to dry by spontaneous evaporation, and it is better to spread the moist mass in an even layer upon paper than to dry it in a heap in the mortar, because it is essential that the drying be uniform, otherwise some portions of the preparation become very hard and dissolve with difficulty in water. A small amount of water must remain in the product in order to render it readily soluble. It should, therefore, be dried only sufficiently to make its pulverization possible, and to prevent its caking together after being bottled.

Description.—A white powder, entirely but rather slowly soluble in 10 parts of water, requiring about 10 minutes to dissolve. The solution is not permanent, however, basic salts soon separating. This decomposition is prevented by the presence of alkali citrates. An unclear solution may be rendered clear by gently heating it, and does not become turbid again for several days.

Varieties of Magnesium Citrate.—Hager states that normal magnesium citrate occurs in three forms, differing from each other in the proportion of water they contain, and in solubility. *Amorphous* magnesium citrate contains 2 to 3 molecules of water, and is soluble in 2 parts of water at ordinary temperatures; *metamorphous* magnesium citrate contains about 5 molecules of water, and is soluble in 10 parts of water; and the *crystalline* magnesium

citrate contains 7 molecules of water, and requires 100 parts of water for its solution. The amorphous variety, when in watery solution, gradually changes to the crystalline form, passing through the metamorphous form, this alteration being usually completed in from three to five days.

The changes referred to are, at least partially, prevented by the presence of alkali-citrates.

Effervescent Magnesium Citrate.

Magnesium carbonate	10 Gm
Citric acid	46 Gm
Sodium bicarbonate	34 Gm
Sugar, in fine powder	8 Gm
Alcohol,	
Distilled water.	

Mix the magnesium carbonate intimately with 30 Gm of citric acid and 4 ml of distilled water, so as to form a thick paste. Dry this at a temperature not exceeding 30° C. and reduce it to a fine powder. Then mix it intimately with the sugar, the sodium bicarbonate, and the remainder of the citric acid previously reduced to a very fine powder. Dampen the powder with a sufficient quantity of alcohol, so as to form a mass, and rub it through a No. 6 tinned-iron sieve. Then dry it, and reduce it to a coarse, granular powder.

Keep the product in well-closed vessels.

Notes. By reference to the preceding notes under the title Magnesium Citrate it will be seen that the first part of the process has for its object the formation of acid magnesium citrate. This is then dried, powdered, and mixed with powdered sugar, sodium bicarbonate and citric acid. The citric acid must be finely powdered. It is necessary that these ingredients should be very thoroughly mixed before the alcohol is added, and only enough alcohol to dampen the mixture should be used. The small amount of water contained in the normal magnesium citrate and in the alcohol is sufficient to cement the particles of powder together into little granules, and the coarse powder is run through a No. 6 sieve to make it more uniform. As a brass sieve would be attacked by the compound, a tinned iron sieve is directed to be employed. The sodium bicarbonate is not decomposed by the citric

acid, in the dry mixture, and hence the preparation, when dissolved in water, yields an effervescing drink.

Description.—A white, coarsely granular mixture, deliquescent on exposure to air, inodorous, having a mildly acidulous taste and acid reaction. Soluble, with copious effervescence, in two parts of water; nearly insoluble in alcohol. The aqueous solution contains sodium-magnesium citrate, some free carbonic acid, citric acid, and sugar.

Solution of Magnesium Citrate.

Contains $MgHC_6H_5O_7$.

Magnesium carbonate	15	Gm
Citric acid	30	Gm
Syrup of citric acid	65	ml
Potassium bicarbonate	2.5	Gm

Dissolve the citric acid in 120 ml of water, add the magnesium carbonate, and stir until dissolved. Filter the solution into a strong bottle of the capacity of 360 ml, and containing the syrup. Then add enough water, previously boiled and filtered, to nearly fill the bottle, drop into it the potassium bicarbonate, and immediately insert the cork, securing it with twine. Lastly, shake the mixture occasionally until the potassium bicarbonate is dissolved.

Reaction. $4MgCO_3.Mg(OH)_2+5H_3C_6H_5O_7=5MgHC_6H_5O_7+6H_2O+4CO_2$.

Notes. Acid magnesium citrate, which is contained in this preparation, does not yield any deposit of magnesium salt such as is formed after about three days in solutions of normal magnesium citrate. As calcium citrate is insoluble in water, the magnesium carbonate and the water used ought perhaps to be free from lime. Acid calcium citrate is, however, soluble to a limited extent. It is directed in the Pharmacopœial working formula that the water added last shall be "previously boiled and filtered." As this precaution has for its object the removal of any lime present, the same treatment should be applied to the whole of the water used, if it be intended that the preparation shall be free from calcium salt, for all of the calcium citrate formed from the calcium car-

bonate present in the water will dissolve in the excess of citric acid used, unless the water employed is quite "hard," in which case it is unfit for use.

The water as well as all other materials used should be perfectly pure. Impure materials lead to the formation, sooner or later, of slimy deposits in the product.

Heat materially hastens the solution of the acid and the magnesium carbonate, and with the quantity of citric acid taken no harm will result if the water in which the acid is dissolved be hot, and the heat continued until the magnesium carbonate has dissolved. The solution should then be filtered, and allowed to become quite *cold* before the other ingredients are added. The clear syrup should be first put in the bottle; then the filtered acid solution of magnesium citrate carefully added so as not to mix it with the layer of syrup covering the bottom of the bottle; then the bottle should be *nearly* filled with water, and the potassium bicarbonate, in large, clear crystals, dropped in, after which the cork is at once driven into the neck of the bottle.

The bottles used for this preparation are made strong enough to bear the pressure caused by the carbonic acid liberated from the potassium bicarbonate by the citric acid. They must necessarily be of such size as to be nearly filled by the solution when finished and of proper strength. The corks used must be about 1½ inches long, and of the finest quality, as large as they can possibly be used, and soaked in hot water so as to be soft and elastic when inserted; the soaked corks ought to be fitted to the necks of the bottles when the latter have been cleaned, but while still empty, and should be so large that, after using the cork presser, they can be driven in by one or two smart blows with a mallet, closing the bottles perfectly. The wire or twine used to tie the corks down securely must be properly attached to the necks of the bottles beforehand, leaving a loop on one side long enough to reach to the center of the top of the cork, and the two ends on the opposite side, so that as soon as the cork has been driven in rather more than one-half its length, it can be at once tied over.

The quantity of potassium bicarbonate used has been advantageously increased to 2.50 Gm, so that the solution may be well charged with carbonic acid. The bottles should be laid on their sides in a cool place, taking care not to shake the contents so as to mix the syrup with the lighter watery solution. The potassium

bicarbonate will then rest in the syrup, and dissolve but slowly. When required for use, the bottle is shaken until all of the bicarbonate is dissolved. Solution of citrate of magnesium is rather agreeable to the taste when properly made, fresh, and cold. It loses its agreeable taste entirely when kept in a warm place, when kept too long, or when taken without first being properly cooled on ice or otherwise.

Magnesium oxide may be employed instead of magnesium carbonate, using only five twelfths as much of the oxide. But the carbonate is more reliable.

MAGNESIUM LACTATE.

MAGNESII LACTAS.

$Mg(C_3H_5O_3)_2+aq.$

Magnesium carbonate	1 part
Lactic acid	2 parts
Distilled water	20 parts

Dissolve the lactic acid in the water, add the magnesium carbonate gradually to the solution gently heated in a porcelain dish over a water-bath. When all the magnesium carbonate has been added and dissolved, filter the solution and evaporate it to crystallization.

Description.—A white, crystalline, odorless, feebly bitter salt; soluble in 26 parts of water.

MAGNESIUM OXIDE.

MAGNESIA; U. S. LIGHT MAGNESIA. LIGHT MAGNESIUM OXIDE.

(Calcined Magnesia.)

$MgO=40.2.$

Light magnesium carbonate.

Rub it through a No. 60 sieve. Heat it in a capacious unglazed earthenware dish, with constant stirring, on a sand-bath, until a sample removed from the center, cooled, mixed with a little water,

and then dropped into diluted sulphuric acid, gives but a very slight effervescence.

Reaction. $(MgCO_3)_4.Mg(OH)_2.(H_2O)_5$ when strongly heated is decomposed into $5MgO+6H_2O+4CO_2$.

Notes. The magnesium carbonate being very light a great deal of it rises from the dish like dust and is scattered about.

In operating upon larger quantities a bright, clean iron pot may be used, placed immediately over the fire and heated gradually to dull redness.

If heated too strongly or too long the magnesia becomes "dead-burnt" so that it does not become "hydrated" and form a gelatinous mass of hydroxide when mixed with water. This failure of the oxide to react with water results whenever the carbonate is heated until all of it is completely decomposed so that no effervescence whatever occurs when the product is tested with diluted sulphuric acid. On the other hand, the product may, if not heated long enough, contain much basic anhydrous carbonate although it gelatinizes with water; in such a case it does not retain the property of becoming converted into gelatinous hydroxide. A *slight* effervescence with diluted sulphuric acid is, therefore, allowable; but considerable effervescence with diluted acid should condemn the product.

Magnesium oxide may also be made in a crucible, packed nearly full of magnesium carbonate with the aid of a pestle, the crucible being covered and heated in an oven or furnace, first moderately and finally to dull redness for about an hour, after which the contents may be tested, and then heated again if necessary. But this procedure is more risky, as the product is very liable to become "dead-burnt" in this way, since it can not be so frequently tested to prevent it.

Magnesia must be kept in small, filled, tightly-corked bottles, the corks dipped in melted paraffin to effectively exclude air so as to prevent the absorption of CO_2 and the consequent formation of carbonate. Containers capable of holding about 50 Gm of magnesia are perhaps the most suitable in size.

Test. When 1 Gm of magnesia ("light magnesia") is thoroughly stirred with 15 ml of water in a beaker and the mixture allowed to stand in the beaker for an hour, a semi-translucent

gelatinous mass of magnesium hydroxide should be formed, which is firm enough to remain in the beaker when the latter is inverted. A magnesia which does not possess the quality to thus form hydroxide is not to be used medicinally, being inferior.

Description.—A white, very light and extremely fine powder; odorless and tasteless, but producing in the mouth the sensation usually produced by dry, absorbent, tasteless, earthy substances. Insoluble in alcohol, and practically insoluble in water, although when moistened it exhibits a slightly alkaline reaction on litmus paper.

"Light magnesia" is about three times as bulky, weight for weight, as "heavy magnesia."

Heavy Magnesia.

MAGNESIA PONDEROSA.

(Heavy Magnesium Oxide.)

$MgO=40.2$.

A white, very fine powder, about three times as dense as the light magnesia, but otherwise of similar physical properties. It also conforms to the reactions and tests given by the Pharmacopœia for "Magnesia," but does not form a gelatinous hydroxide with water, or does so only with great difficulty.

MAGNESIUM SALICYLATE.

MAGNESII SALICYLAS.

$Mg(C_7H_5O_3)_2.4H_2O=370.2$.

Salicylic acid	14 parts
Distilled water	200 parts
Magnesium carbonate, sufficient.	

Mix the salicylic acid and distilled water in a large porcelain dish and heat the mixture over a water-bath. Add 5 parts of magnesium carbonate to the hot liquid, stir well, and continue heating until effervescence ceases.

Test a filtered portion of the liquid on litmus paper, and if the

reaction is acid add more magnesium carbonate, enough to render the reaction of the liquid almost entirely neutral.

When the liquid shall be almost or quite neutral to test-paper, let it cool, filter, and then add enough salicylic acid to render the reaction distinctly acid.

Filter again, and evaporate to crystallization.

Notes. In order to obtain a product which will keep well and produce a clear solution with water, it is necessary to render the liquid acid from an excess of salicylic acid before evaporating to crystallization.

The magnesium carbonate employed should be free from iron in order to insure a perfectly colorless product.

As a supersaturated solution may be easily formed it is necessary to determine the right degree of concentration of the liquid by repeated trials during the process of evaporation.

The fine crystalline powder deposited while the liquid is stirred during the process of cooling, is to be collected, the crystals carefully freed from adhering mother-liquor, and dried.

Additional crops of crystals may be obtained by evaporation of the mother-liquor, but the crystals thus obtained are usually more or less colored (reddish).

Description.—Colorless crystals, permanent in the air, odorless, of sweetish-bitterish taste. Soluble in 10 parts of water, forming a clear solution which has a distinctly acid reaction. Soluble in alcohol.

MAGNESIUM SULPHATE.

MAGNESII SULPHAS.

(Epsom Salt.)

$MgH_2SO_5.6H_2O=246.2.$

Magnesite, in powder	20 parts
Sulphuric acid (92.5% of H_2SO_4)	25 parts
Water	70 parts

Mix the magnesite in a porcelain dish with 40 parts of water. Then add about 2 parts of the sulphuric acid, and stir. When effervescence has ceased add another

portion of the acid, and again wait until the reaction has subsided. Continue adding sulphuric acid in portions in the same manner until about two-thirds of it has been used. Then add about 20 parts of water, and heat the mixture to about 90°. Add the remainder of the acid, in portions, as before; then add the rest of the water, and heat the whole at about 90° for two hours, stirring frequently. Add enough magnesium carbonate to render the solution neutral to litmus paper. Set the liquid aside to cool and settle. Filter. Evaporate the filtrate until it has about 1.33 sp. w. while still hot, and then set it aside to crystallize. Drain the crystals and dry them at about 25°.

Reaction. $MgCO_3+H_2SO_4=MgSO_4+H_2O+CO_2$.

Description.—Small, colorless, acicular crystals; odorless; taste cooling, saline, bitter. Slightly efflorescent in dry air. Soluble in 1.5 parts of water at 15° and in 0.7 part of boiling water. Insoluble in alcohol.

Purified Magnesium Sulphate.

Dissolve any desired quantity of commercial Epsom salt in one and one-half times its weight of water, filter the solution and set it aside to crystallize.

Should the Epsom salt contain iron the solution should be digested with a little magnesium carbonate before crystallization. *Large* crystals are readily obtained if the crystallization is slow.

Dried Magnesium Sulphate.

MAGNESII SULPHAS EXSICCATUS.

$MgH_2SO_5.H_2O=156.2$.

Expose crystallized magnesium sulphate in a porcelain dish to the heat of the water-bath until the salt ceases to lose weight.

Notes. Crystallized magnesium sulphate effloresces somewhat in dry air and at from 30° to 52° loses one molecule of water. The dried salt, on the other hand, absorbs water from a moist air. The product must, therefore, be kept in tightly closed bottles.

MAGNESIUM SULPHITE.

MAGNESII SULPHIS.

$MgSO_3.6H_2.O=212.2.$

By saturating either magnesium carbonate or magnesium oxide, suspended in water, with sulphurous oxide, the crystalline product being drained and dried.

Description.—A white, crystalline powder; odorless; taste bitterish, somewhat sulphurous. Soluble in 20 parts of water.

Must be kept in tightly-stoppered bottles, as it oxidizes on exposure to air.

MAGNESIUM TARTRATE.

MAGNESII TARTRAS.

$MgC_4H_4O_6=172.2.$

Tartaric acid	10 parts
Magnesium carbonate	6 parts

Dissolve the acid in 20 parts of distilled water, keep the solution hot over a water-bath, and add gradually the magnesium carbonate until a neutral salt is obtained. Evaporate to dryness, and powder the residue.

Description.—A white powder which is practically insoluble in cold water.

MANGANESE CARBONATE.

MANGANI CARBONAS.

$MnCO_3=115.$

Manganous sulphate	22 parts
Sodium bicarbonate	17 parts

Dissolve the bicarbonate in 200 parts of boiling water; add the

sulphate previously dissolved in 100 parts of boiling water and sweetened with 5 parts of sugar. Wash the precipitate with boiling water containing about 4 per cent of sugar. When the washings give but a slight turbidity with test solution of barium chloride, press the water out of the magma, and dry it as rapidly as practicable, with the aid of moderate heat.

Description.—A dirty white powder with a reddish tinge. It is insoluble in water, but dissolves in dilute acids.

MANGANESE DIOXIDE.

MANGANI DIOXIDUM.

(Black Oxide of Manganese.)

MnO_2=87.

Pure manganese dioxide is not obtainable. The best grades of pyrolusite or brownstone contain about 90 per cent of MnO_2. The impurities in native black oxide of manganese are ferric oxide, silica, barium carbonate, calcium carbonate, alumina, etc. The American Pharmacopœia requires the manganese dioxide to contain not less than 66 per cent of MnO_2. Since this is the case the official title should not be "manganese dioxide," but "native black oxide of manganese."

In the employment of the native mineral its variable composition must be taken into account. In the trade the pyrolusite occurs both whole and powdered. The whole mineral can not be adulterated; but the powdered is frequently mixed with coal, etc.

Extremely fine powder is necessary for some purposes; but for the preparation of chlorine from hydrochloric acid a coarsely powdered or crushed pyrolusite is the best.

The Pharmacopœia describes it as a heavy, grayish-black, more or less gritty powder, without odor or taste. Insoluble in water, alcohol, or any other simple solvent. Not affected by cold sulphuric or hydrochloric acid.

MANGANESE CHLORIDE.

MANGANI CHLORIDUM.

$MnCl_2.4H_2O$=197.8.

The acid liquid containing manganese chloride which remains in the flask or other generator when chlorine gas is prepared from hydrochloric acid and manganese dioxide can be used to make manganous chloride as follows:

Evaporate the liquid to dryness and continue the heat as long as acid vapors pass off. Boil the residue with water for about ten minutes. Filter the solution. Precipitate manganous carbonate from about one-eighth of the filtrate with an excess of sodium carbonate solution; wash the precipitate and then add it to the other seven-eighths of the filtrate. Boil for about half an hour, adding more water if required. Filter, evaporate, and crystallize.

The preparation may be readily made from manganous sulphate and barium chloride.

Description.—Pale red, deliquescent crystals, soluble in alcohol as well as water.

MANGANESE IODIDE SYRUP.

SYRUPUS MANGANI IODIDI.

Manganous sulphate	40 Gm
Potassium iodide	50 Gm
Sugar	280 Gm
Simple syrup, distilled water, each sufficient.	

Dissolve each of the salts in 50 ml of water mixed with 10 ml of simple syrup. Mix the solutions and shake well. Cool the mixture to about 10° C. (50° F.). Filter, rinsing the precipitate on the filter with a little sweetened water, letting the filtrate and washings run into a bottle containing the sugar. Shake until dissolved, and then add enough distilled water to make the total product measure 400 milliliters.

Must be protected from light.

Reaction. $MnSO_4+2KI=K_2SO_4+MnI_2$.

Notes. The manganous iodide is very deliquescent and prone to rapid decomposition. Hence it is not used except preserved by sugar in the form of syrup.

The application of cold in the process is to cause the separation of the potassium sulphate as far as possible.

Description.—A clear, pale, rose-colored or pinkish syrup. It becomes nearly colorless on standing if exposed to light, the sugar becoming partially inverted at the same time.

MANGANESE PHOSPHATE.

MANGANI PHOSPHAS.

$Mn_3(PO_4)_2.4H_2O=427$.

Manganous sulphate	10 parts
Sodium phosphate	11 parts

Dissolve the salts, separately, each in 80 parts of hot water, filter, and let the solutions cool. Mix them. Wash the precipitate with water until the washings cease to give any reaction for sulphate. Dry the product with the aid of gentle heat, avoiding unnecessary exposure to the air.

Reaction.

$$3(MnSO_4.4H_2O)+2(Na_2HPO_4.12H_2O)$$
$$=Mn_3(PO_4)_2.4H_2O+2(Na_2SO_4.10H_2O)+H_2SO_4+12H_2O.$$

Notes. As will be seen, the reaction is analogous to that occurring in the preparation of ferroso-ferric phosphate. Loss of product is sustained by reason of the solubility of manganous phosphate in the sulphuric acid formed. The free acid may be carefully neutralized by the addition of sodium bicarbonate to obviate this loss.

Description.—An almost white (slightly pinkish) powder, odorless and tasteless; insoluble in water and in alcohol.

MANGANOUS SULPHATE.

MANGANI SULPHAS.

$MnSO_4.4H_2O=223.$

Manganese dioxide.
Concentrated sulphuric acid.
Sodium carbonate.
Water.

Mix the finely powdered manganese dioxide with a sufficient quantity of strong sulphuric acid to form a very soft paste. Heat this mixture in a porcelain dish, strongly, on a sand-bath, stirring constantly, until dry. Then transfer the dry mass to a Battersea crucible and keep it at a red heat for about an hour. Let cool. Leech out the salt with hot water, filter the solution.

Set aside seven-eighths of this solution. To the remaining eighth add a solution of sodium carbonate (1 part of carbonate in 10 parts of water) as long as a precipitate falls. Wash this precipitated manganous carbonate by decantation until the washings are free from sulphate.

Then add the washed magma of manganous carbonate to the reserved portion of the solution of manganous sulphate, mix well, and heat the mixture in a porcelain dish to boiling.

Filter, and evaporate the filtrate to crystallization.

Collect, drain, and dry the crystals.

Bottle the product at once as soon as the crystals are dry to the touch.

Reaction. $2MnO_2+2H_2SO_4=2MnSO_4+2H_2O+O_2.$

Notes. The solution of manganous sulphate obtained from one kilo of manganese dioxide may be evaporated to about 900 Cc. and then set aside in a shallow dish, over sulphuric acid, in a desiccator, when a solid cake of crystals will be obtained in a few days, at the ordinary room temperature. These crystals, however, contain 5 molecules of water. The Pharmacopœia orders a salt with 4 molecules of water, and this can be obtained by redissolving the manganous sulphate obtained as described in an equal weight of water and letting this solution stand in a dish at 30° until crystallization

takes place. At a very low temperature the salt crystallizes with 7 molecules of water.

When the crystals are drained they should be covered with a damp piece of cloth to prevent efflorescence.

The object of adding manganous carbonate to the solution of manganous sulphate is the precipitation and removal of any iron, which may still remain after the ignition which decomposes nearly all of the iron sulphate without decomposing the manganese salt.

Description.—Pale rose-colored, or colorless; transparent crystals; odorless; taste slightly bitter, astringent. Efflorescent in dry air. Soluble in 0.8 part of water at 15°, and in 1 part of boiling water. Insoluble in alcohol.

MERCURY; PURIFIED.

HYDRARGYRUM PURIFICATUM.

Hg=200.

Mercury	10 parts
Nitric acid	1 part
Water	5 parts

Put the metal with the acid and water in a porcelain dish and let them remain together during four days, stirring the contents frequently and strongly. Then pour off the acid liquid, and wash the metal, first with water and finally with distilled water, until the washings no longer give an acid reaction on litmus paper. Dry the mercury by pouring it through a cone made of white filter paper or blotting paper and having an aperture at the apex barely large enough to admit a pin, so that the metal passes through in a thin stream.

Notes. Common mercury usually contains one or more of the metals, lead, tin, copper, antimony, arsenic, bismuth. Such impure mercury sometimes presents a dull surface. Drops of mercury containing lead do not retain their globular form when made to roll about on white paper, but leave streaks or traces and may even form little tail-like projections.

Nitric acid attacks the tin, antimony and some of the other metals before it attacks the mercury and removes them, leaving

the mercury purer than before, and sufficiently pure for the preparation of the mercury compounds to be employed for pharmaceutical purposes.

Mercury boils at 357.°25 C. (U. S. P.), and the most effective method of purification of commercial mercury is distillation.

Mercury should present a perfectly bright surface, but it may present a dark colored film or patches on the surface if contaminated with dust, dirt, moisture, or the oxides of the foreign metals present. To restore its brightness it is only necessary to run the metal through a paper cone as before described; but if other metals are present the mercury becomes dull and dirty again from metallic oxides floating on its surface.

MERCURIC BENZOATE.

HYDRARGYRI BENZOAS.

$$Hg(C_6H_5CO_2)_2=442.$$

Freshly precipitated mercuric oxide from 27 parts of mercuric chloride is mixed with 22.5 parts of artificial benzoic acid (from toluol) and the mixture digested at a temperature near the boiling point (at full water-bath heat) until the yellow color of the mercuric oxide has given place to the yellowish-white color of the mercuric benzoate formed. The product forms voluminous masses floating in the liquid.

Let the liquid cool to 50° C., transfer the whole mixture to a cloth strainer or a paper filter, according to the quantity operated upon, and wash the benzoate with water of the temperature of from 50° to 60° C.

Dissolve the salt in boiling water and let the solution cool so that the product may be obtained in crystals. Dry these at from 40° to 50° C.

Description.—Mercuric benzoate thus prepared is in colorless, glistening crystal needles, of a somewhat metallic taste. Gives an acid reaction on moist blue litmus paper. Nearly insoluble in cold water, but freely soluble in boiling water. Readily soluble in a water solution of sodium chloride, and this solution has been used for hypodermatic injection. Soluble in alcohol but with partial decomposition. It contains about 45.2 per cent of Hg.

MERCURIC BROMIDE.

HYDRARGYRI BROMIDUM.

$HgBr_2$=360.

Mercury	5 parts
Bromine	4 parts
Water	25 parts

Place the metal in a flask; add the water; then the bromine. Apply heat, gradually increased, until the mercury is dissolved. Boil, filter, and then crystallize.

Reaction. $Hg+2Br=HgBr_2$.

Notes. The action is at first slow, but when some $HgBr_2$ has been formed the bromine dissolves more freely and the reaction goes on rapidly.

If necessary, a little more bromine should be added to complete the solution of the last of the mercury.

Description.—A white, odorless, crystalline solid; soluble in 20 parts of water at 15°.

MERCURIC CHLORIDE.

HYDRARGYRI CHLORIDUM CORROSIVUM.

(Corrosive Sublimate. Corrosive Chloride of Mercury.)

$HgCl_2$=270.8.

Mercuric sulphate	20 parts
Sodium chloride	15 parts
Manganese dioxide	1 part

Mix the dried and powdered materials and triturate the mixture thoroughly until a uniform and very fine powder is obtained. Put this into a suitable vessel and heat in a sand-bath until the mercuric chloride has been sublimed.

Reaction. $HgSO_4+2NaCl=Na_2SO_4+HgCl_2$.

Notes. The materials must be thoroughly dry and reduced to very fine powder. The manganese dioxide should be strongly heated after having been powdered and before it is mixed with the other substances.

On a small scale the same apparatus as described in the notes under the head of Hydrargyri Chloridum Mite may be employed. Or a retort may be used having the neck cut off rather short so as to leave a wide opening. The retort should be buried in the dry sand up to the neck. Heat is then applied gradually. If the materials were not perfectly dry water vapor will be formed, and it is then necessary to heat gently at first until the moisture shall have been expelled, and even the neck of the retort should be warmed sufficiently to insure the expulsion of all water vapor. The iron dish employed for the sand-bath is then heated more and more strongly until sublimation is effected, the end of the neck of the retort being capped with a wide-mouthed bottle. The heat is continued until the sublimation is completed, care being taken not to permit the neck of the retort to become choked up. Towards the close of the process the heat is raised almost to the fusing point of the mercuric chloride in order to obtain a sublimate of more decidedly developed crystalline structure. The retort is finally allowed to cool slowly, after which the sublimate may be readily removed from the short neck.

In order to obviate the danger of inhaling any vapor of mercuric chloride it is necessary that the whole operation be carried out in a hood with a good draught. The product must not be carelessly handled, being extremely poisonous. If it is desired to powder corrosive sublimate, it should be kept dampened with alcohol during the process of trituration, to prevent the rising of dust.

Should the mercuric sulphate employed contain any mercurous sulphate a corresponding quantity of mercurous chloride must be produced. To prevent this the manganese dioxide is added. The rationale of its action is shown in the following equation:

$$Hg_2SO_4 + MnO_2 + 2NaCl = Na_2SO_4 + 2HgO + MnCl_2.$$

Without the addition of manganese dioxide it is scarcely possible to obtain a corrosive sublimate that does not contain calomel

to such an extent as to produce a very unclear solution in water. But the sublimed mercuric chloride may be dissolved in diluted alcohol and crystallized from the filtered solution.

Another Method.

Mercuric oxide.....	9 parts
Hydrochloric acid (32% of HCl).....	10 parts
Distilled water.....	500 parts

Heat together until the oxide is completely dissolved. Filter the hot solution, and evaporate it to dryness. Crystallize the product.

Reaction. $HgO+2HCl=HgCl_2+H_2O$.

Notes. The product obtained by this method may be sublimed to obtain it in the form of crystals or crystalline masses, or it may be crystallized in needles from a solution made with warm diluted alcohol, or with water.

Solutions of mercuric chloride for certain purposes may be readily made of definite strength from mercuric oxide, hydrochloric acid and water in such proportions as may be required. The hydrochloric acid is prescribed in slight excess.

Description.—Heavy, transparent, colorless, odorless crystals or crystalline masses, having an acrid, nauseous, persistent, metallic taste. It is extremely poisonous, and should, therefore, not be tasted except in the most cautious manner.

Obtained by sublimation on a large scale, or heated at or near its fusing point (about 265°) and then very slowly cooled, it forms large, transparent, colorless crystals. When crystallized from solutions it is obtained in the form of long needles which are somewhat opaque. Soluble in 16 parts of water, and in 3 parts of alcohol, at 15°; in 2 parts of boiling water, and in 1.2 parts of boiling alcohol; also in 4 parts of ether, and in 14 parts of glycerin.

Mercuric chloride dissolves so slowly in water that in making solutions of it the most advantageous procedure is to employ powdered chloride and hot water.

MERCURIC CHLORAMIDE.

HYDRARGYRUM AMMONIATUM.

(Mercurammonium Chloride. Ammoniated Mercury. White Precipitate.)

$H_2NHgCl=251.4$.

Mercuric chloride..... 10 parts
Ammonia water, distilled water, each sufficient.

Dissolve the chloride in 200 parts of warm distilled water; filter and allow to cool. Pour the filtrate gradually and during constant stirring into 5 parts of ammonia water, taking care that the ammonia shall remain in excess when all of the mercuric chloride has been added. Collect the precipitate at once on a filter, drain as much as possible, and then wash it twice, each time with a mixture of 20 parts of distilled water and 1 part of ammonia water. Finally, dry the precipitate between filter paper in a dark place at a temperature not exceeding 30° C. (86° F.).

Reaction. $HgCl_2+2H_4NOH=H_4NCl+H_2NHgCl+2H_2O$.

Notes. When a solution of mercuric chloride is poured into ammonia water, the latter being in excess throughout, the official ammoniated mercury is formed.

When, however, the order of mixing the liquids is reversed, the ammonia water being added to the mercuric chloride, or when the mercuric chloride is present in excess over the proportion of ammonia used, the product will be, or will contain, $H_2N(HgCl)_2Cl$ instead of H_2NHgCl. The $H_2N(HgCl)_2Cl$ is, however, converted into the official mercurammonium chloride when allowed to remain in contact with an excess of ammonia for some time.

Some pharmacopœias order the addition of the ammonia to the solution of mercuric chloride; others, including ours, direct the opposite order of mixing.

Mercurammonium chloride is perfectly white. A grayish color indicates that the solution of mercuric chloride was not filtered before adding it to the ammonia, whereby the calomel,

which is always present in corrosive sublimate, gives rise to mercurous oxide. If warm liquids are used, or if the precipitate be washed with warm water or too long, or if ammonia is not present in the washing and drying, the product will be more or less yellow from hydroxyl-dimercurammonium chloride ($HNOHHg_2Cl$). The same result follows if a too high temperature is used in drying. Exposure to light renders it grayish.

The preparation must be kept in well closed bottles, in a cool place, and protected from light.

MERCURIC CYANIDE.

HYDRARGYRI CYANIDUM.

$Hg(CN)_2=252.$

Potassium ferrocyanide..... 60 parts
Solution of ferric chloride (U. S.)..... 83 parts
Mercuric oxide.
Water.

Dissolve the potassium ferrocyanide in 600 parts of hot water, and filter. Dilute the solution of ferric chloride with 750 parts of water. Pour the solution of potassium ferrocyanide gradually into the solution of ferric chloride, stirring constantly. Set the mixture aside until the precipitate has subsided as far as it may. Decant the supernatant liquid. Add to the magma about 1,000 parts of boiling water acidulated with 10 parts of hydrochloric acid, stir well, and let the mixture stand until the ferrocyanide of iron has again settled. Decant, and add another 1,000 parts of boiling water, acidulated as before; mix thoroughly; let settle, and decant again. Repeat the washing of the precipitate at least twice again in the same manner. Then mix the blue magma well with 1,000 parts of boiling water (this time *not* acidulated), let settle, and decant. Transfer the wet mass to a muslin strainer and continue the washing with pure boiling water until the washings are no longer rendered turbid by test-solution of silver nitrate. When the washing has been completed enclose the magma in the strainer and express the water from it as far as possible. Dry the iron ferrocyanide in a porcelain dish with the aid of moderate heat.

Triturate 1 part of this ferrocyanide of iron with 2 parts of mercuric oxide until thoroughly mixed. Put the mixture in a porcelain dish, add 20 parts of water, and heat upon a water-bath for one hour, stirring frequently and replacing the water lost by evaporation. Then boil the mixture until the blue color disappears. Should a blue color still remain after boiling the mixture for ten minutes, add a little more mercuric oxide and continue boiling until the color of the insoluble matter becomes black.

Filter the liquid. Boil the black precipitate with about 10 parts of water, filter, and add this liquid to the other filtrate. Supersaturate the united filtrates with hydrocyanic acid, using enough to impart a permanent odor of that acid to the liquid.

Now filter the solution and evaporate to crystallization.

Collect the crystals in a funnel, let drain, and then dry them on a filter paper with the aid of moderate heat.

Evaporate the mother-liquor to obtain additional crystals in the usual way.

Reactions.

First, $3K_4Fe(Cy)_6+4FeCl_3=Fe_4(FeCy_6)_3+12KCl$; and then

$$Fe_4(FeCy_6)_3+9HgO+9H_2O$$
$$=9HgCy_2+3Fe(OH)_2.4Fe(OH)_3.$$

Notes. The precipitated iron ferrocyanide contains potassium compound and the wash water is acidulated with hydrochloric acid to remove that compound. In order to render the precipitate more dense, to facilitate the otherwise extremely difficult washing, boiling water is repeatedly employed. The washing is completed with pure water to remove the hydrochloric acid as well as the last traces of potassium chloride. To facilitate the drying of the ferrocyanide the water is forcibly pressed out, and the finely divided "prussian blue" is then spread out thinly over the bottom and inner sides of a large porcelain dish and moderate heat used.

To make the mercuric cyanide from ferrocyanide of iron, the latter must be recently precipitated and of the right composition. Hence the commercial "prussian blue" can not be successfully employed.

The solution of mercuric cyanide obtained by boiling ferro-

cyanide of iron with mercuric oxide contains some basic salt ("oxy-cyanide of mercury"), and it is, therefore, necessary to acidulate this solution with hydrocyanic acid. Some iron usually precipitates on this addition of hydrocyanic acid.

The hydrocyanic acid required for this purpose may be made from potassium cyanide. A sufficient quantity for the product obtained from 60 parts of potassium ferrocyanide will be produced by 4 parts of potassium cyanide which may be dissolved in 80 parts of diluted alcohol and mixed with 9.2 parts of tartaric acid after which the mixture is allowed to stand for one hour and then filtered.

Another Method.

Potassium cyanide	26 parts
Tartaric acid	60 parts
Mercuric oxide	43 parts
Diluted alcohol	500 parts
Water.	

Dissolve the potassium cyanide in the diluted alcohol. Add the powdered tartaric acid and shake well. Let the mixture stand one hour, shaking occasionally. Filter in a covered funnel into a bottle. Add the mercuric oxide to the filtrate and let stand, shaking frequently, until the odor of hydrocyanic acid has nearly ceased and the mercuric oxide has dissolved. Evaporate the filtered solution to crystallization, after first adding some more hydrocyanic acid.

Reactions.

First, $KCy+H_2C_4H_4O_6=KHC_4H_4O_6+HCy$; then

$HgO+2HCy=HgCy_2+H_2O$.

Notes. Should the solution of mercuric cyanide be basic it deposits mercuric oxide on evaporation. It is, therefore, necessary to have the hydrocyanic acid in excess. It is best to dissolve the mercuric oxide and then add enough hydrocyanic acid to impart its odor to the liquid. (See notes under preceding formula.)

Description.—Colorless or white crystals; odorless; of bitter metallic taste. It is extremely poisonous, and, therefore, should

not be tasted except in a diluted condition and with the utmost caution.

It is darkened by exposure to light and must, therefore, be kept in dark amber-colored bottles or in a dark place.

Soluble at 15° in 12.8 parts of water, and in 15 parts of alcohol; in 3 parts of boiling water and in 6 parts of boiling alcohol.

MERCURIC IODIDE.

HYDRARGYRI IODIDUM RUBRUM.

(Red Iodide of Mercury.)

HgI_2=453.

Mercury	4 parts
Iodine	5 parts
Alcohol.	

Triturate the iodine in a glazed porcelain mortar (or in a glass mortar) with 2 parts of alcohol. Add the mercury. Continue the trituration until a uniform scarlet red powder has been obtained, adding a little more alcohol from time to time as may be necessary to keep the mixture moist until the chemical reaction shall have been accomplished as far as practicable.

Reaction. $Hg+I_2=HgI_2$.

Notes. A pure mercuric iodide is not obtained by this method for the reaction is not complete. The alcohol is added to prevent the vaporization of any of the iodine by the heat generated by the reaction, the alcohol being evaporated instead of the iodine and the temperature thereby reduced.

This method is not recommended, but is included in this manual only as an illustration of the preparation of chemical products by dry trituration.

The product obtained from 4 parts of mercury and 5 parts of iodine may be put in a flask with 160 parts of alcohol and dissolved by boiling; the hot solution may then be poured into a beaker and allowed to cool slowly in order to obtain the mercuric iodide in crystals.

Wet Process.

Mercuric chloride, in powder.....	12 parts
Potassium iodide.....	15 parts

Dissolve the mercuric chloride in 200 parts of hot distilled water, and filter. Dissolve the potassium iodide in 40 parts of cold distilled water, and filter.

Pour the solution of mercuric chloride, when cold, slowly, into the solution of potassium iodide, stirring constantly. Set aside until the precipitate has subsided perfectly. Decant the mother liquor and reject it. Wash the precipitate with 300 parts of cold water, stirring it up well, allowing it to subside again, and at once decanting the washings. Then collect the precipitate upon a white paper filter, and continue washing with distilled water until the washings cease to give a precipitate with test-solution of silver nitrate.

Dry the iodide between white filter paper at a temperature not exceeding 40° C. (104° F.). Keep it in well closed bottles.

Reaction. $HgCl_2+2KI=HgI_2+2KCl$.

Notes. Theoretically we should require for 12 parts of mercuric chloride about 14.7 parts of potassium iodide; but the potassium iodide generally contains small quantities of carbonate and other impurities, and it is moreover desirable to use a slight excess of the potassium iodide, to prevent the formation of a light-red compound of mercuric chloride with mercuric iodide. For this reason, also, the solution of mercuric chloride must be poured into that of the potassium iodide so as to keep the latter at all stages of the process in excess over the mercuric chloride with which it is being mixed, and constant stirring is directed with the same object in view.

Unless the solutions are filtered before mixing them, the product, besides retaining mechanical impurities, may possibly also contain mercurous chloride, the mercuric chloride being frequently contaminated with calomel.

Crystallized mercuric iodide. Use the same materials and in the same proportions as given above, but use hot solutions. The mercuric iodide being soluble in a hot solution of potassium chloride, the product crystallizes in the gradual cooling.

Being soluble in 15 parts of boiling alcohol, the mercuric iodide may be easily crystallized from a hot alcoholic solution.

Description.—A scarlet-red, heavy, odorless and tasteless powder. Amorphous if made by triturating mercury and iodine together, or by cold precipitation; crystalline if made by precipitation with hot solutions. Brilliant, although small, crystals can be obtained from a hot alcoholic solution.

Soluble in 130 parts of alcohol at 15°, and in 15 parts of boiling alcohol. Soluble also in solutions of potassium iodide, mercuric chloride, and sodium thiosulphate, respectively.

Becomes yellow when heated to about 150°, but turns scarlet again on cooling. It fuses at about 238° to a dark-yellow liquid which forms a mass of minute yellow crystals on cooling, but these crystals turn red again after a time (or at once, if triturated).

MERCURIC NITRATE SOLUTION.

LIQUOR HYDRARGYRI NITRATIS.

A liquid containing about 60 per cent of mercuric nitrate ($Hg(NO_3)_2$=324) together with about 11 per cent of free nitric acid.

Red mercuric oxide	8 parts
Nitric acid	9 parts
Distilled water	3 parts

Mix the acid and water and dissolve the oxide in the mixture.

Keep the solution in glass stoppered bottles.

Reaction. $HgO+2HNO_3=Hg(NO_3)_2+H_2O$.

Description.—A clear, colorless, heavy, corrosive liquid, strongly acid in its reaction, and having an odor of nitric acid. The sp. w. is about 2.100 at 15° C.

British Solution of Mercuric Nitrate.

The "liquor hydrargyri nitratis acidus" of the British Pharmacopœia is prepared as follows:

Mercury	400 Gm
Nitric acid	500 ml
Distilled water	150 ml

Mix the acid and water in a flask. Dissolve the mercury in the mixture without the aid of heat. Then boil gently for fifteen minutes, or until a drop of the liquid no longer gives any precipitate in dilute hydrochloric acid. Let it cool, and preserve the solution, which should weigh about 1,200 Gm, in a stoppered bottle away from the light.

Reaction. $3Hg+8HNO_3=3Hg(NO_3)_2+4H_2O+2NO.$

Nitrate of Mercury Ointment.

UNGUENTUM HYDRARGYRI NITRATIS.

(Citrine Ointment.)

Mercury	14 parts
Nitric acid (68%)	35 parts
Lard oil	152 parts

Heat the lard oil in a porcelain dish to a temperature of 100° C.; remove the dish from the source of heat; gradually add to the oil 14 parts of nitric acid, and, when the effervescence caused by the reaction begins to subside, heat the dish again to 60° until the reaction is completed and effervescence has ceased. Then allow the mixture to cool down to about 40° C.

Dissolve the mercury in 21 parts of nitric acid with the aid of sufficient heat to prevent the solution from crystallizing, and add this liquid to the warm mixture made of lard oil and nitric acid as above described. Stir well, and then let the mixture stand until cold, after which it must be again mixed thoroughly in a porcelain mortar or dish with a pestle of the same material, using a spatula of horn, wood, or porcelain to remove the ointment from the mortar and pestle.

Notes. The reactions occurring when fat is treated with strong nitric acid are not well known; however, the lard oil is acted upon with more or less energy according to the temperature, and the olein is converted by oxidation at the expense of the nitric acid into elaidin, which is a firmer fat, having a fusing point of about 32° C. It is necessary that the nitric acid used should be of full strength in order that this reaction may be completed before the solution of mercuric nitrate is added. If the temperature is perfectly controlled, the acid used of full strength, and the reaction

thoroughly completed before the nitrate of mercury is added, the product will be a handsome, firm, lemon yellow ointment. If the heat is too high, the mercury nitrate will be decomposed and the product rendered brown, or gray from mercury oxide and reduced metal.

In mixing the mercuric nitrate solution with the oxidized fat, it is necessary to stir well with a porcelain spatula or glass rod until nearly cold, in order to thoroughly incorporate the heavy liquid which might otherwise settle to the bottom of the capsule.

The solution of mercuric nitrate to be added to the ointment may be advantageously made by dissolving mercuric oxide (instead of the metal) in nitric acid.

MERCURIC OLEATE.

HYDRARGYRI OLEAS.

$Hg(C_{18}H_{33}O_2)_2$=762.

Mercuric oxide	30 Gm
Nitric acid (1.42 sp. gr.)	26 Gm
Solution of potassium oleate	1,500 ml

Mix the oxide, the acid, and 200 ml of distilled water, in a beaker or an evaporating dish, and heat the mixture until the oxide dissolves, adding a few drops more of nitric acid, *if necessary.* Dilute the solution of mercuric nitrate thus formed with three liters of water. Add 1,500 ml of solution of potassium oleate (prepared as described under that title). Warm the mixture and wash the separated mercuric oleate twice with warm distilled water, using about one liter each time. Subject the oleate to a low temperature in a mortar, and then press out of it all the water by means of a pestle.

Reaction.

$$Hg(NO_3)_2+2KC_{18}H_{33}O_2=Hg(C_{18}H_{33}O_2)_2+2KNO_3.$$

Notes. Mercuric chloride cannot be employed in place of the nitrate, nor can sodium oleate be used instead of the potassium oleate, as the washing then becomes very difficult.

The yield is about 100 Gm or a little more. The product is a soft, yellow or reddish yellow solid, and contains 28.4 per cent of mercuric oxide.

The official "oleate of mercury" (U. S. P.), is a soft solid made of 1 part of mercuric oxide and 4 parts of oleic acid. This oleate is best prepared at not over 40° C., the little lumps of oxide being carefully broken down by rubbing between two horn or wooden spatulas, two or three times a day, until the oxide is perfectly dissolved, which requires 8 or 10 days. An oleate containing 20 per cent of oxide keeps fairly well.

When small quantities are made the oxide and oleic acid may be mixed in a porcelain mortar with the pestle.

Mercuric oleate is very sensitive to heat, decomposing with the separation of finely divided, dark colored, metallic mercury. Precipitated oleate of mercury containing no free oleic acid keeps better than solutions of mercuric oleate in free oleic acid, and the semi-solid "20 per cent oleate" keeps better than the liquid "10 per cent oleate" (containing 10 per cent of oxide).

Official Oleate of Mercury.

Yellow mercuric oxide, thoroughly dried..	200 Gm
Oleic acid.....	800 Gm

Introduce the oleic acid into a capacious mortar, and gradually add to it the yellow mercuric oxide by sifting it upon the surface of the acid, and incorporate it by continuous stirring. Then set the mixture aside in a warm place, at a temperature not exceeding 40° C. and stir frequently, until the oxide is dissolved.

Description.—A soft, light brownish-yellow ointment-like solid.

MERCURIC OXIDE; RED.

HYDRARGYRI OXIDUM RUBRUM.

HgO=216.

Dissolve 8 parts of mercury in 9 parts of official nitric acid previously diluted with 4 parts of water. Evaporate the solution

to dryness. To the residue add another 8 parts of mercury, and triturate in a mortar until metallic globules are no longer visible and the mixture appears to be uniform. Heat this mixture in a porcelain dish over a sand-bath, stirring frequently, until acid vapors are no longer evolved.

Reactions.

First, $3Hg+8HNO_3=3Hg(NO_3)_2+4H_2O+2NO$.

Second, $Hg(NO_3)_2+Hg=Hg_2(NO_3)_2$.

Third, $Hg_2(NO_3)_2$, when heated, decomposes into

$$2HgO+N_2O_4.$$

Notes. The nitrate is decomposed by heat, red fumes (nitrogen tetroxide) being evolved. When all the nitrate has been thus decomposed, the acid vapors cease, and the residue is mercuric oxide. Care must be taken to continue the heating until not a trace of nitrate remains. Mercuric oxide when strongly heated becomes very dark, but the red color returns on cooling. If the heat is too high (over 360° C.) the oxide decomposes into its elements, which may be discovered by holding a piece of filter-paper over the heated mass, when metallic mercury will condense on the paper.

It is sometimes directed to digest the residue after cooling, with distilled water to which a little solution of soda has been added, the object being to remove any remaining nitrate. In this case the oxide must afterwards be thoroughly washed, and dried at a gentle heat.

Description.—A brick-red, insoluble powder. Must not produce red vapors when heated. It is completely soluble in HCl, producing $HgCl_2$.

Another Method.

A finely divided red mercuric oxide may be obtained by heating the yellow (precipitated) mercuric oxide until it becomes red on cooling, and corresponds to the pharmacopœial identity tests.

MERCURIC OXIDE; YELLOW.

HYDRARGYRI OXIDUM FLAVUM.

[Precipitated Mercuric Oxide.]

$HgO=216$.

Mercuric chloride	100 Gm
Sodium hydroxide	40 Gm
Distilled water, sufficient.	

Dissolve the powdered mercuric chloride in 3 liters of hot distilled water, and filter the solution.

Dissolve the sodium hydroxide in 500 ml distilled water.

Pour the solution of mercuric chloride, when cold, slowly and in a small stream, or a little at a time, into the solution of sodium hydroxide, stirring briskly and uninterruptedly.

Allow the mixture to stand for an hour, stirring frequently.

Decant the supernatant liquid from the precipitate, and wash the latter by affusion and decantation of cold distilled water.

Collect the precipitate on a strainer or a paper filter and continue the washing with warm distilled water until a portion of the washings when poured over a little test-solution of mercuric chloride no longer produces a yellowish turbidity at the point of contact of the two liquids.

Then allow the precipitate to drain, and dry it between sheets of white blotting paper, in a dark place, at a temperature not exceeding 30° C.

Keep the product in well-stoppered bottles in a dark place.

Reaction. $HgCl_2+NaOH=HgO+2NaCl+H_2O$.

Notes. The mercuric chloride is used in the form of powder and hot water is to be used to dissolve it in order to save time. But the solution of mercuric chloride must be cold when poured into the solution of sodium hydroxide. The use of very hot solutions in making precipitated mercuric oxide would yield a product consisting partly of the red variety. The use of a too high heat in washing and drying yellow oxide of mercury is to be avoided for the same reason. An over-heated freshly precipi-

tated mercuric oxide has a more reddish color than the product not exposed to a too high temperature, and the reddish precipitated oxide does not readily form mercuric oxalate when treated with a strong solution of oxalic acid as described in the Pharmacopœia.

As mercuric chloride is liable to contain calomel, the omission of the filtration might result in the discoloration of the product by the mercurous oxide formed by that calomel.

The sodium hydroxide used should contain at least 90 per cent of NaOH, and must be free from carbonate. The presence of sodium carbonate in the "soda" used would cause the formation of mercuric carbonate, which is dark brown and usually falls after the orange yellow mercuric oxide has subsided and thus forms a thin layer over the oxide.

Dilute solutions are employed because the use of a strong solution of mercuric chloride might easily lead to the formation of "oxychloride of mercury" having a brown or brick-red color. There are two such oxychlorides, one of them so dark brown as to appear nearly black ($HgCl_2.2HgO$), and the other brown-red or brick-red ($HgCl_2.3HgO$). In order to prevent the formation of either of these oxychlorides it is necessary that the mercuric oxide formed should not be permitted to come in contact with the mercuric chloride. Hence the mercuric chloride must be completely decomposed as fast as it is added to the solution of sodium hydroxide. This result is insured by (1) making the solution of mercuric chloride very dilute while the solution of sodium hydroxide is less diluted; (2) adding the solution of the mercuric chloride to the solution of sodium hydroxide, instead of *vice versa;* (3) adding the mercury solution very gradually; (4) stirring constantly and briskly while mixing the solutions; and using a larger amount of sodium hydroxide than required by theory according to the chemical equation, so that the alkali shall be in excess from beginning to end.

Should the solution of sodium hydroxide be poured into that of the mercuric chloride, the mercuric oxide then formed would at once (and throughout the process of mixing the solutions) come into contact with the mercuric chloride, and would then form oxychloride, which would be evidenced by the dark, dirty color of the precipitate.

Should mercuric carbonate or any oxychloride be formed, caus-

ing the precipitate to appear "off color," they may be decomposed by maceration with an excess of sodium hydroxide. Hence the precaution to "let the mixture stand for an hour."

But if the mercuric oxide is contaminated with oxychloride or with carbonate it usually requires a stronger solution of sodium hydroxide and longer time than an hour to insure their complete decomposition and the final formation of a pure, bright orange-yellow mercuric oxide.

Strict observance of all the directions here given as to the degree of dilution of the solutions, the order and manner of mixing them, and the purity and proportions of the materials, will always insure a satisfactory result.

The washing is to be continued until the washings are free from alkali. The washings may be tested for sodium hydroxide in the manner prescribed, or for sodium chloride by slightly acidulating with nitric acid and then using test-solution of silver-nitrate.

Light affects mercuric oxide comparatively rapidly, decomposing it into metallic mercury and oxygen. Precipitated mercuric oxide, being exceedingly finely divided, is more rapidly decomposed than the red mercuric oxide. The metallic mercury formed by the reduction is also extremely finely divided and hence appears black, thus darkening the product. Hence the light should be entirely excluded from mercuric oxide.

Yellow or precipitated mercuric oxide may also be made from an acid solution of mercuric nitrate instead of the mercuric chloride, and potassium hydroxide may be used in the place of sodium hydroxide.

Description.—A fine, orange-yellow powder, amorphous, very heavy, odorless, and of a somewhat metallic taste. Insoluble in water and in alcohol. Darkens on exposure to air, decomposing into mercury, mercurous oxide, and oxygen.

MERCURIC PEPTONATE SOLUTION.

LIQUOR HYDRARGYRI PEPTONATI.

1. Make a solution of 1 part of mercuric chloride in 20 parts of distilled water.

2. Dissolve 3 parts of dry peptone (free from sodium chloride) in 10 parts of distilled water.

3. Dissolve 1 part of sodium chloride in 50 parts of distilled water.

Pour solution 1 slowly and with constant stirring into solution 2. Let the mixture stand an hour. Collect the precipitate on a filter and let it drain well. Transfer the precipitate to a porcelain dish, add solution 3 and stir well until solution results. Then add enough distilled water to make the whole product weigh 100 parts. Filter.

Keep it in small, completely filled and well-stoppered bottles in a cool, dark place.

Description.—A yellowish, acid liquid of disagreeable metallic taste.

MERCURIC SALICYLATE.

HYDRARGYRI SALICYLAS.

$HgC_7H_4O_3$=336.

Mercuric chloride	27 parts
Solution of sodium hydroxide (5%)	250 parts
Salicylic acid	15 parts
Distilled water, sufficient.	

Dissolve the mercuric chloride in 540 parts of hot, distilled water, filter the solution, and let it cool to the ordinary room temperature.

Dilute the sodium hydroxide solution with 50 parts of distilled water.

Pour the cold solution of mercuric chloride gradually and during uninterrupted stirring into the solution of sodium hydroxide. Wash the precipitate by decantation and finally on a paper filter with distilled water until the washings give no further reaction for chloride.

Collect the precipitate and put it in a flask with enough distilled water to form a thin mixture when shaken. Then add the salicylic acid, shake again so that the mixture may be made as nearly uniform as practicable, and then heat the flask on a water-bath, shaking the flask frequently.

When the yellow color of the mercuric oxide has given place to the pure white color of the mercuric salicylate, which is the finely

divided insoluble matter now contained in the liquid, remove the flask from the water-bath.

Wash the mercuric salicylate with hot water until the washings no longer give an acid reaction on test-paper. Then collect the product on a paper filter, let it drain well, and dry it, at first at a moderate heat and finally at 100° C.

Notes. The salicylic acid is used in slight excess and forms mercuric salicylate with the freshly precipitated mercuric oxide. The excess of salicylic acid is then washed out.

Description.—A white, amorphous, odorless and tasteless powder, almost insoluble in water and in alcohol.

Mercuric salicylate is not decomposed by carbonic acid, or by tartaric, lactic or acetic acid, nor by sodium hydroxide solution. When the salt is dissolved in a solution of sodium hydroxide in such proportion that one molecule of NaOH is present for each molecule of $HgC_7H_4O_3$, a double salt is formed which crystallizes out of the solution.

MERCURIC SULPHATE.

HYDRARGYRI SULPHAS.

$HgSO_4$=296.

Mercury	100 Gm
Sulphuric acid	60 ml

Heat them together in a porcelain capsule, stirring constantly, until the metal disappears and a dry white salt remains.

Reaction. $Hg+2H_2SO_4=HgSO_4+SO_2+2H_2O$.

Notes. Mercury is insoluble in cold sulphuric acid.

Mercuric sulphate is used in the preparation of calomel, corrosive sublimate, and basic mercuric sulphate.

It may also be made, as described in the next paragraph, with the aid of nitric acid, when less sulphuric acid will be required.

Description.—A heavy white crystalline powder which decomposes on the addition of water, yielding yellow subsulphate, or

Basic Mercuric Sulphate.

HYDRARGYRI SUBSULPHAS FLAVUS.

[Yellow Subsulphate of Mercury. Turpeth Mineral.]

$(HgO)_2HgSO_4 = 728.$

Mercury	100 Gm
Sulphuric acid	30 ml
Nitric acid	25 ml
Distilled water, sufficient.	

Put the mercury in a roomy flask, add the sulphuric acid, previously mixed with 15 ml of distilled water, and then the nitric acid, previously mixed with 25 ml of distilled water. Digest the mixture at a gentle heat until red fumes cease to be given off. Transfer the mixture to a porcelain dish and heat it on a sand-bath, under a hood or in the open air, with frequent stirring, until a dry, white salt remains. This white salt is normal mercuric sulphate. Powder this and throw it, in small portions at a time, into 2 liters of boiling distilled water, stirring constantly. Having added all of the mercuric sulphate, boil the mixture ten minutes, allow it to settle, decant the liquid, transfer the precipitate to a filter, wash it with hot distilled water until the washings cease to give an acid reaction on test-paper, and finally dry the product in a moderately warm place.

Reaction. $2Hg+2H_2SO_4+2HNO_3=2HgSO_4+3H_2O+N_2O_3$. Then the mercuric sulphate is split up by water into a basic salt which precipitates, and an acid salt which remains in solution, the result varying somewhat according to the quantity of water used and its temperature.

Notes. Mercuric sulphate can be made from mercury and sulphuric acid without the use of nitric acid; when the latter is added, however, the reaction is more easily accomplished.

Basic mercuric sulphate can also be made by gradually adding a solution of mercuric nitrate to a solution of sodium sulphate, the latter salt to be in excess.

Description.—A heavy, lemon-yellow powder, odorless and nearly tasteless, very slightly soluble in water, insoluble in alcohol

or ether. When heated it turns red, but becomes yellow on cooling.

MERCURIC SULPHIDE; RED.

HYDRARGYRI SULPHIDUM RUBRUM.

(Vermillion.)

$HgS=232$.

Mercury	150 parts
Sulphur	57 parts
Potassium hydroxide	38 parts
Water.	

Triturate the mercury with the sublimed sulphur until no more globules of mercury are visible under a lens of ten diameters magnifying power.

Dissolve the potassium hydroxide in 300 parts of water. Add the black mercuric sulphide formed by the trituration of the metal with the sulphur. Heat at 45° in a porcelain dish for several hours, stirring briskly at frequent intervals, and replace the water lost by evaporation so as to maintain the original volume. When the powder assumes a bright scarlet color, turn the contents of the dish into a suitable vessel and wash the sulphide quickly by affusion and decantation of warm water (having a temperature not exceeding 45°), until the washings are tasteless and quite neutral to test-paper. Collect the product on a strainer or filter, drain, and dry it at from 45° to 50°.

Reactions. $Hg+S=HgS$; also $8KOH+4S=3K_2S+K_2SO_4+4H_2O$.

Notes. The quantity of sulphur prescribed in this formula (Brunner) is much in excess of that accounted for by the foregoing reactions; the excess is dissolved by the solution of potassium sulphide, perhaps forming hypothiosulphite and tetrathiosulphate.

It is not understood why the black mercuric sulphide made by trituration becomes bright scarlet when the excess of sulphur is removed from it by digestion with the potassium hydroxide.

The temperature must not exceed 45°.

As continued trituration of the mercuric sulphide with the solution of potassium hydroxide hastens the change of color of the product, this may be accomplished in the same mortar in which

the black sulphide was made, provided the mortar is large enough. It should be kept warm by placing it in warm water.

The yield will be about 100 parts.

Crude Mercuric Sulphide (Mixed with Sulphur).

(Æthiops Mineralis.)

Mercury	1 part
Sulphur	1 part

Triturate them together in a glass mortar (or in a glazed porcelain mortar) until a uniform black powder shall have been obtained in which no globules of mercury are visible under a lens of ten diameters magnifying power.

Reaction. $Hg+S=HgS$.

Notes. As the atomic weight of mercury is 200, and that of sulphur only 32, it follows that this product consists of about 3 parts of mercuric sulphide and 2 parts of sulphur. It is an old preparation formerly employed in veterinary medicine, and is included in this book simply as an illustration of chemical combination between dry solids triturated together.

The trituration must be continued at least two hours under strong pressure. This necessarily causes the formation of shining black "scales," which must finally be reduced to powder by gentler trituration and by sifting the product through a fine sieve.

The mortar and pestle are at the end of the trituration covered with a black coating of mercuric sulphide which is not easily removed if the mortar used be one made of wedgewood ware or other porous material. Strong nitric acid and powdered glass or white sand may be triturated together in the mortar to remove the stain.

MERCUROUS CHLORIDE.

HYDRARGYRI CHLORIDUM MITE.

(Calomel. Mild Mercurous Chloride.)

$HgCl=235.2$.

Mercuric sulphate	10 parts
Mercury	7 parts
Sodium chloride, dried	5 parts
Boiling distilled water, sufficient.	

Moisten the mercuric sulphate with some of the water; add the mercury; triturate until metallic globules are no longer visible; add the sodium chloride and mix the whole thoroughly by long continued trituration. Sublime by a suitable apparatus. Wash the sublimate with boiling distilled water until the washings cease to be darkened by a drop of ammonium hydrosulphide. Dry it at not over 100° C.

Reaction. $HgSO_4+Hg=Hg_2SO_4$; then, $Hg_2SO_4+2NaCl=2HgCl+Na_2SO_4$.

Notes. On a small scale the sublimation may be effected in bottles of about 200 Cc. capacity. These bottles should be thin-bottomed and of uniform thickness, and should have rather wide mouths. During the process of sublimation the mouths of the bottles may be closed loosely with chalk stoppers. The mixed powder to be subjected to sublimation must be thoroughly dried before it is put into the bottles. Flasks or retorts may also be used. The bottles, flasks, or retorts are heated by means of a sand-bath. They should be only about one-fourth filled and buried into the sand sufficiently deeply to bring the level of the contents of the subliming vessel below the level of the sand surrounding it. The heat must be applied cautiously, being moderate at first, then gradually increased until the calomel has all been sublimed.

Sublimed calomel is yellowish-white when prepared in small quantities and in the way here described. When manufactured on a large scale, the vapor is rapidly condensed in large chambers into which steam is injected, and the product is then nearly or quite white and more finely divided.

The sublimate obtained in operating with small amounts is crystalline and must be triturated with warm water a long time (levigated) until reduced to an extremely fine (impalpable) powder. The warm water used for this purpose is to be frequently changed in order that the mercuric chloride may be effectually removed from the calomel.

The heat must not be too high for then a portion of the calomel is decomposed into mercury and mercuric chloride.

The sublimate formed in vessels heated in a sand-bath is deposited immediately above the level of the sand as well as higher up. To remove the sublimate it is necessary to break the vessel

containing it. Great care should be exercised in doing this so that no fragments of glass become mixed with the product. If necessary to detach the crust from the glass while the sublimate is still warm, it is best to moisten it a little; but after a few days the crust can be readily removed without being moistened.

All sublimed calomel, however made, is contaminated with mercuric chloride, so that calomel free from corrosive sublimate can be obtained only by levigating the sublimed or precipitated calomel until pure. Elutriation may be advantageously combined with the levigation. When the water with which the calomel is levigated ceases to become darkened by ammonium hydrosulphide or to be rendered unclear on the addition of ammonia, and when the wet calomel fails to produce a dark, dull spot on polished steel after five minutes' contact with it the product is free from mercuric chloride.

Another Method.

Mercuric chloride	4 parts
Mercury	3 parts
Alcohol.	

Triturate the mercuric chloride and mercury together in a porcelain mortar until all metallic globules have disappeared, keeping the mixture moistened all the time with some alcohol to prevent the poisonous dust from rising. When no more mercury globules are visible the mixture is gray. Put the gray mixture, still damp from the alcohol used, in a porcelain dish and heat it moderately upon a sand-bath, with constant stirring, until the moisture, mercuric chloride, and mercury have been expelled, leaving a yellowish-white nearly pure calomel, which will be the case when a white sublimate is formed on the bottom of a flask or bottle held immediately above the contents of the dish when strongly heated. This part of the process must be performed under a hood with a good draft, or in any manner insuring the removal of the poisonous vapors which pass off. The calomel mixture, thus thoroughly dried and partially freed from mercuric chloride, is then put in bottles, flasks, or retorts, and sublimed as described in the notes under the preceding process.

Description.—A heavy, white, impalpable powder; odorless and tasteless. Becomes yellowish-white on being triturated under

strong pressure. Should show only isolated crystals under a magnifying power of one hundred diameters. Insoluble in all simple solvents. Volatilizes without residue.

When exposed to light it turns grayish from finely divided metallic mercury, mercuric chloride being at the same time formed. Calomel must, therefore, be kept in dark amber-colored bottles, or otherwise protected from the light.

Precipitated Mercurous Chloride. F

HYDRARGYRI CHLORIDUM MITE PRAECIPITATUM.

(Precipitated Calomel.)

Mercury ..... 175 parts
Nitric acid, 68% ..... 150 parts
Distilled water, sodium chloride, and hydrochloric acid.

Put the mercury in a tared flask; add 100 parts nitric acid and 150 parts water. Heat the flask moderately on a sand-bath, covering the mouth of the flask with a watch-glass, and continue the heat without interruption for one hour after red fumes have ceased to be evolved, or until the liquid begins to acquire a yellowish color and to become slightly turbid. Pour the solution of mercurous nitrate off from the undissolved mercury.

Mix 50 parts of nitric acid with 1000 parts of boiling distilled water, and add this mixture to the solution of mercurous nitrate while still hot. Let the mixture cool.

Test the liquid to ascertain whether or not it will bear dilution with an equal volume of cold distilled water without becoming turbid. To perform this test mix about 5 ml of the solution with 5 ml of distilled water. Should the mixture remain clear, return it to the whole quantity of the mercury solution. Should it become turbid, add a sufficient quantity of nitric acid to the whole solution to acidify it so that dilution with twice its volume of water without precipitation is rendered assured.

Ascertain the quantity of mercury contained in the solution by deducting from the quantity originally taken the weight of the undissolved portion remaining in the flask. [That undissolved mercury must be washed and dried before weighing it.]

Take a quantity of sodium chloride equal to the weight of the dissolved mercury; dissolve that sodium chloride in 20 times its

weight of distilled water, and add to this solution 5 per cent of its weight of hydrochloric acid. Then pour the sodium chloride solution gradually into the mercury solution, stirring constantly.

Wash the precipitate with cold distilled water, by affusion and decantation, until the washings no longer give an acid reaction on test-paper.

Dry the product at a temperature not exceeding 40° C.

Reactions. $3Hg+4HNO_3=3HgNO_3+2H_2O+NO$, and subsequently $HgNO_3+NaCl=HgCl+NaNO_3$.

Notes. It is necessary that the mercury solution should contain enough nitric acid to remain clear when diluted with its own volume of water, and it is to be tested to make sure of that fact before the solution of sodium chloride is added, because if the mercury solution should contain too little nitric acid the precipitate formed upon dilution with water is a basic mercurous nitrate and this would then be precipitated by the solution of sodium chloride together with the calomel. But if the solution of mercurous nitrate will bear dilution with its own volume of water without becoming turbid, it may safely be mixed with more than its own volume of sodium chloride solution, gradually added, without any danger of the precipitation of basic mercurous nitrate together with the mercurous chloride.

The large dilution of the liquids is necessary to prevent the formation of mercuric salts by the nitric acid.

Precipitated calomel is much bulkier than sublimed calomel, and is extremely finely divided.

It should be tested for nitrate, from which it must be quite free.

Another Method.

Dissolve 1 part of mercuric chloride in 50 parts of distilled water. Keep the solution at a temperature of from 70° to 80° C., and conduct into it a current of washed sulphur dioxide (prepared from sodium bisulphite and diluted sulphuric acid as described under the title of sulphurous acid, until saturated with it. Let the liquid cool. Wash the precipitated calomel upon a filter with distilled water. Dry the product with the aid of gentle heat.

Reaction. $2HgCl_2+SO_2+2H_2O=2HgCl+H_2SO_4+2HCl$.

Description.—See sublimed calomel. The only differences between sublimed calomel and precipitated calomel is that the latter is lighter (bulkier), being finer, and less liable to contain crystals (visible under the microscope, only).

Mercurius Solubilis Hahnemanni.

[Nitras amido-hydrargyricus cum hydrargyro; Danish Ph.]

Mercurous nitrate	180	parts
Nitric acid	12.8	parts
Ammonia water	100	parts
Alcohol	1150	parts

Distilled water, sufficient.

Dissolve the mercurous nitrate in a cold mixture of the nitric acid with 1800 parts of distilled water. Pour this solution slowly into a mixture of the ammonia water and alcohol, stirring constantly.

[The mixture thus obtained should have a feebly acid reaction.]

Collect the precipitate on a filter, let it drain, and wash it with a small quantity of alcohol. Dry it in a dark place and without the aid of heat.

Must be protected from the light.

Description.—A heavy, dead black, fine powder, odorless, tasteless, insoluble in water and in alcohol. Heated to a high temperature it decomposes, giving off nitrous vapors, and leaves no fixed residue. Heated with sodium hydroxide solution it gives off ammonia. Should be completely soluble in nitric acid.

MERCUROUS IODIDE.

HYDRAGYRI IODIDUM FLAVUM.

Yellow Iodide of Mercury.

$HgI = 326.5$.

Mercury	8	parts
Iodine	5	parts

Alcohol.

Triturate the iodine in a glazed porcelain mortar (or in a glass

mortar) with three parts of alcohol. Add the mercury. Continue the trituration until all globules of mercury have disappeared and the mixture, when nearly dry, has acquired a greenish-yellow color. During the trituration, add more alcohol, from time to time, as may be necessary to keep the mixture constantly moist.

When the reaction is at an end, as indicated by the disappearance of mercury globules and by the development of a greenish-yellow color, add enough alcohol to reduce the whole to a thin mixture, liquid enough to be poured into a bottle. Use sufficient additional small portions of alcohol to rinse the mortar well and to transfer all of the wet powder to the same bottle.

Cork the bottle and set it aside in a dark but moderately warm place for a day, shaking it occasionally during that period. Then let the heavy powder subside completely, pour off the alcohol from the solid matter and add, instead of the alcohol thus decanted, about 50 parts of pure alcohol, warm the bottle, shake well, and again set it aside, well corked, for a day or two.

Decant again, and, if necessary, repeat once more the washing of the mercurous iodide with another portion of warm alcohol.

Finally shake the bottle well, transfer its contents to a white paper filter, and continue washing the product on the filter with warm alcohol until the washings are no longer darkened by hydrogen sulphide or by a weak solution of sulphurated potassa.

When the washing shall have been completed as described, dry the product between white blotting paper in a dark place at a temperature not exceeding 40°.

Keep the product in a well-closed bottle and protected from light.

Reaction. $Hg+I=HgI$.

Notes. The reaction between the two elements causes much heat. The alcohol added before and during the trituration is evaporated by this heat, and thus keeps the temperature down so as to prevent the loss of iodine by vaporization, which would be sustained were the materials triturated together in a dry state. But the reaction is not complete. The product obtained by this method contains mercury and mercuric iodide as well as mercurous iodide. The mercury cannot be removed, and its presence gives the mercurous iodide a greenish hue. Pure mercurous iodide is

yellow without greenish tint. The mercuric iodide is washed out with warm alcohol.

The alcohol used for washing out the mercuric iodide may all be recovered by distillation after adding some solution of potassium hydroxide.

The best method for the preparation of mercurous iodide is by the decomposition of mercurous nitrate with potassium iodide.

Another Method.

Mercury ..... 4 parts
Mercuric iodide ..... 9 parts
Alcohol, sufficient.

Triturate the metal with the mercuric iodide, keeping the mixture constantly moist with alcohol, until no more mercury globules can be detected with a lens of ten diameters magnifying power. Wash the greenish-yellow powder by digestion with warm alcohol until the washings are no longer darkened by hydrogen sulphide.

Reaction. $HgI_2+Hg=HgI$.

Notes. Compare this method with the one immediately preceding it.

Official Method; U. S. P.

Mercury ..... 50 Gm
Nitric acid,
Potassium iodide,
Distilled water,
Alcohol.

Mix 20 ml, each, of nitric acid and distilled water, and, when the liquid is cold, pour it upon the mercury contained in a small glass flask. Set the mixture aside in a cool and dark place, and agitate it occasionally, until the reaction ceases, and a little mercury still remains undissolved. Separate the crystals of mercurous nitrate, which will have formed, from the mother-liquid, allow them to drain in a glass funnel, and dry them on bibulous paper, in a dark place. When the salt is dry, weigh off 40 Gm of it, and dissolve it in 1 liter of distilled water to which 10 ml of nitric acid had previously been added. Having prepared a solution of 24 Gm of potassium iodide in 1 liter of distilled water, slowly pour the

solution of potassium iodide into that of the mercurous nitrate, with constant stirring, allow the precipitate to subside, decant the supernatant liquid, and transfer the precipitate, together with the remainder of the liquid, to a filter. When the precipitate has drained, wash it with distilled water until the washings no longer have an acid reaction upon litmus paper, and afterwards wash it with alcohol, as long as the clear, colorless washings give any color with hydrogen sulphide test-solution. Lastly, dry the product in a dark place, between sheets of bibulous paper, at a temperature not exceeding 40° C.

Keep it in dark, amber-colored bottles, with the least possible exposure to light.

Reactions. $3Hg+4HNO_3=3HgNO_3+2H_2O+NO$; and, then,

$HgNO_3+KI=HgI+KNO_3$.

Notes. The preparation of mercurous nitrate is described elsewhere. Instead of adding the mercury nitrate to the potassium iodide, which would cause the precipitation of some basic mercurous nitrate with the mercurous iodide, it is necessary that the usual order of mixing the reagents be reversed in this case; hence the formula directs that the solution of potassium iodide be added to the solution of mercurous nitrate. The precipitated mercurous iodide is light yellow. It should be protected against light throughout the process of preparation as well as afterwards. The mercuric iodide which is formed with the mercurous iodide, owing to the presence of mercuric nitrate and nitric acid with the mercurous nitrate, is washed out with alcohol.

Instead of weighing off 40 Gm of the mercurous nitrate as above directed, the whole of the crystallized salt may be taken and the amount of potassium iodide, etc., adjusted in accordance with the proportions given above.

Description.—A bright yellow, amorphous, odorless, tasteless, insoluble, heavy powder. Darkens on exposure to light, decomposing into mercuric iodide and mercury.

Made by triturating mercury and iodine, or mercury and mercuric iodide, together, the preparation is invariably yellowish-green because the reaction is not complete so that the product contains mercuric as well as mercurous iodide and mercury.

MERCUROUS NITRATE.

HYDRARGYROSUS NITRAS.

$HgNO_3.H_2O=280.$

Add a cold mixture of 30 parts of nitric acid and 20 parts of distilled water to 55 parts of mercury, in a flask. Let the mixture stand in a dark place, agitating it occasionally, until reaction ceases and but little of the metal remains undissolved. Collect in a glass funnel the crystals of mercurous nitrate which have been formed in the cold liquid, and allow them to drain. Then place the crystals on white blotting paper and dry them in a dark place.

Reaction. $3Hg+4HNO_3=3HgNO_3+2H_2O+NO.$

Notes. The mercury nitrate remaining in the mother liquid should be recovered as well as the undissolved mercury. The rationale of the process is the formation of normal mercurous nitrate in solution in a mixture of nitric acid and water, employing so small a quantity of liquid that the greater part of the salt crystallizes out.

By digestion of an excess of mercury with the acid and water, or with the acid solution of mercurous nitrate, a basic mercurous salt would soon be formed, which separates in pale yellow crystals or powder. An experienced operator may employ digestion and continue it until basic mercurous nitrate begins to deposit, after which the solution should be decanted, a little nitric acid added to it, and the clear liquid set aside in a cold place that crystals may be formed.

Mercury dissolves in nitric acid with greater or less rapidity according to the temperature and the degree of concentration of the acid. When strong nitric acid is used the solution will contain mercuric nitrate, which, however, is reduced to mercurous nitrate by digestion with more mercury.

By dissolving mercury in cold dilute nitric acid, mercurous nitrate is obtained.

The acid should be present in excess, because otherwise basic salts are formed which are insoluble in the liquid.

Normal mercurous nitrate may be obtained from the solution in colorless plates which can be dissolved in a small quantity of

water, but which decompose if much water is added, an acid mercurous nitrate remaining in solution whilst a yellow basic mercurous nitrate precipitates.

A dilute solution of mercurous nitrate can therefore not be made without an excess of nitric acid.

Description.—Colorless crystals, which partially effloresce on exposure to air, and, when heated, first fuse and then volatilize without residue. Soluble without decomposition in a small quantity of warm water; easily soluble in diluted nitric acid. A larger proportion of water decomposes the salt, causing the formation of a light yellow basic mercurous nitrate.

Tests. A solution of the salt in water strongly acidulated with nitric acid gives a black precipitate with solution of sodium hydroxide, and a white precipitate with hydrochloric acid.

When five hundred milligrams of the salt is triturated with two hundred and fifty milligrams of sodium chloride and water is added, the filtrate obtained from this mixture is darkened on the addition of hydrogen sulphide, but should not give a precipitate with that reagent (absence of mercuric nitrate).

MERCUROUS OXIDE.

HYDRARGYRI OXIDUM NIGRUM.

Hg_2O=416.

Prepared by macerating freshly precipitated mercurous chloride with solution of potassium hydroxide in excess. The thoroughly washed and still moist mercurous chloride is gradually added to the solution of potassa, with diligent stirring; the whole is well shaken, and then set aside in a dark place until the black mercurous oxide has settled. This is then washed with cold distilled water, collected, and dried between folds of blotting paper in a cool, dark place.

Must be kept well protected from the light.

About 12 parts of precipitated calomel, and 24 parts of solution of potassium hydroxide of 1.33 specific weight, will be required to prepare 10 parts of mercurous oxide.

Solution of sodium hydroxide may be used instead of the solution of potassium hydroxide.

MERCUROUS TANNATE.

HYDRARGYRI TANNATUM.

Mercuous nitrate	50 parts
Tannic acid	30 parts
Distilled water, sufficient.	

Triturate the dry mercurous nitrate in a porcelain mortar until reduced to as fine powder as possible.

Triturate the tannic acid with 50 parts of distilled water to a uniform and smooth mixture.

Add the tannin mixture to the powdered mercurous nitrate and triturate together until a perfectly uniform soft mass is obtained, entirely free from hard particles. Then add gradually during constant trituration enough distilled water to produce a thin mixture and pour that into 3,000 parts of distilled water. Wash the greenish precipitate with cold water by decantation and finally on a strainer or on a paper filter until the washings are free from nitric acid. Dry the precipitate on filter paper spread upon porous tiles, or on several thicknesses of white blotting paper, and finally by the aid of heat not exceeding 40° C.

Must be protected from light.

Notes. The composition of the product is not definite, but it is believed to contain *about* 50 per cent of Hg.

At temperatures above 40° C. the moist precipitate is liable to cake together.

It must be well dried in order to keep.

Description.—Brownish-green masses, odorless, tasteless, insoluble in water and in alcohol.

OXYGEN.

OXYGENIUM.

O_2=32.

Potassium chlorate	10 parts
Manganese dioxide	3 parts
Sodium carbonate	2 parts

Fuse the sodium carbonate in its own water of crystallization; add the potassium chlorate; mix well; evaporate to dryness, and powder the mass. Mix this powder intimately with the powdered manganese dioxide. Put the mixture in a suitable flask provided with delivery-tube and safety tube, and heat the contents over a Bunsen burner. The generator or flask should be connected with a wash-bottle containing water.

The reaction is: $2KClO_3 = 2KCl + 3O_2$.

Notes. The manganese dioxide causes the potassium chlorate to decompose more quietly and at a lower temperature than would be the case when the chlorate is heated alone. The sodium carbonate also facilitates the regularity of the decomposition of the chlorate and prevents the formation of gaseous chlorine oxides which are liable to contaminate the oxygen. As a further precaution the gas is washed by passing it through water.

The manganese dioxide must be free from carbon (powdered coal) and organic matter, and the materials perfectly dry.

The retort, Fig. 120, p. 184, may be used.

One kilogram of $KClO_3$ should furnish about 240 liters of oxygen.

One cubic-decimeter of oxygen at 0° C., bar. 760 mm, weighs about 1.43 Gm, so that 1 Gm of oxygen under those conditions occupies the volume of about 699 cubic-centimeters.

PHOSPHORUS.

Phosphorus for medicinal and pharmaceutical uses should be *white*. It is preserved in glass-stoppered bottles filled with water, which should be put in a place where there is no danger that they may be broken.

Phosphorated Oil.

OLEUM PHOSPHORATUM; U. S.

Introduce about 100 Gm of almond oil into a flask and heat it for fifteen minutes over a sand-bath at a temperature of 250° C. Allow it to cool to about 60° C., and then filter it. Put 90 Gm of this filtered oil into a dry and tared bottle of about 120 Cc capacity. Add 1 Gm of phosphorus previously well

dried by gently touching it with white blotting paper. Close the bottle with a glass stopper and heat it in a water-bath until the phosphorus melts. Then shake until the phosphorus has dissolved, allow it to cool, and add enough ether to make the total contents of the bottle weigh 100 Gm. Shake again.

Keep the product in glass-stoppered bottles of about 30 Cc capacity, completely filled, and put in a cool and dark place.

Notes. Keeping the oil of almond at the high temperature prescribed for fifteen minutes expels moisture and air from it, and also causes the separation of some organic matters, which deposit in the form of a flocculent sediment on cooling and are then filtered out. The oil thus prepared is nearly colorless.

The phosphorus used should be clean, translucent pieces.

The ether used should be of the official strength; ordinary commercial ether would render the preparation unclear.

At a too low temperature the phosphorus crystallizes out; this should be carefully guarded against, and when it occurs it should not escape observation. The separated phosphorus may be readily redissolved by gently warming and shaking.

The preparation is not phosphorescent in the dark.

PLATINUM CHLORIDE.

CHLOROPLATINIC ACID.

$$H_2PtCl_6.6H_2O=516.4.$$

Dissolve platinum scraps in a mixture of 1 part of strong nitric acid and 5 parts of strong hydrochloric acid. Evaporate to dryness. Dissolve the residue in hot water, adding a little hydrochloric acid. Add an excess of sodium hydroxide solution. Boil the mixture for about an hour, adding a few drops of alcohol. Redissolve the precipitate in hydrochloric acid, filter, and add a hot saturated solution of ammonium chloride as long as it causes precipitation of ammonium-platinum chloride (or ammonium chloroplatinate). Collect the yellow precipitate and wash it with cold and very dilute hydrochloric acid. Then dry it in a porcelain dish or on glass. Heat it in a porcelain crucible until reduced. When no more ammonia or chlorine is evolved, dissolve the metal again in a mixture of strong hydrochloric and nitric

acids, in the same proportions as before, evaporate to dryness, adding hydrochloric acid from time to time until all nitric acid shall have been expelled. Redissolve the residue in hydrochloric acid and evaporate to crystallization.

Notes. The object of treating the first solution with sodium hydroxide at boiling heat is to reduce any iridium tetrachloride present to trichloride, which afterwards forms a soluble double-salt with ammonium chloride when the ammonium chloroplatinate is precipitated. The alcohol added during the treatment with NaOH is to decompose any sodium hypochlorite formed.

The evaporation to crystallization may be advantageously completed over concentrated sulphuric acid or lime in a desiccator.

Description.—Chloroplatinic acid is a yellow deliquescent salt, soluble in alcohol as well as in water. It is considered as formed by the direct union of platinum tetrachloride and hydrochloric acid.

POTASSIUM ACETATE.

POTASSII ACETAS.

$KC_2H_3O_2=98.$

Acetic acid	5 parts
Potassium bicarbonate	3 parts

Add the potassium bicarbonate gradually to the acid; filter; evaporate over a sand-bath to dryness, stirring constantly, adding a little acetic acid if necessary to keep it in decided excess to the end.

Must be kept in tightly corked bottles.

Reaction. $KHCO_3+HC_2H_3O_2=KC_2H_3O_2+CO_2+H_2O.$

Notes. Theoretically the proportions given in the foregoing formula leave the acetic acid slightly in excess; but this excess of acid will be driven off during the evaporation, and more must be added from time to time as may be required in order to maintain a slightly acid reaction.

The temperature during the evaporation should be 120° to 140° C.

Second Method.

Another method of preparation is as follows:

Lead acetate	13 parts
Potassium bicarbonate	7 parts

Dissolve each salt in about 40 parts of water, filter, and add the solution of lead acetate to that of the potassium bicarbonate, stirring well. Let settle. Decant the supernatant liquid which contains the potassium acetate, add enough acetic acid to it to render it slightly acid, and evaporate to dryness.

Collect and wash the precipitated lead carbonate.

Reaction.

$$3Pb(C_2H_3O_2)_2+6KHCO_3$$
$$=2PbCO_3.Pb(OH)_2+6KC_2H_3O_2+2H_2O+4CO_2$$

Description.—A white powder or feathery crystalline salt of satiny lustre. Odorless. Taste saline, warming. Very deliquescent. Soluble in 0.36 part of water and in 1.9 parts of alcohol at 15°.

POTASSIUM ANTIMONATE.

POTASSII ANTIMONAS.

Metallic antimony	1 part
Potassium nitrate	2 parts

Powder each substance separately. Mix them well. Put the mixture, a small portion at a time, in a crucible heated to dull redness. When all has been added continue the heating for half an hour after the completion of the detonation of the mixture, taking care not to allow the contents of the crucible to fuse. Let cool. Powder the mass, wash the powder well with distilled water, and then dry the product.

Notes. When antimony is strongly heated with potassium nitrate the metal decomposes the nitrate and a salt is formed which was at one time official in some pharmacopœias under the title of "superstibias kalicus," or acid potassium antimonate.

If the mixture of the antimony and potassium nitrate, after detonation, should be heated to so high a temperature that the mass undergoes fusion, the product is not the same, being water-soluble.

The composition is uncertain.

Description.—A white or but slightly yellowish powder, insoluble in water. [If the antimony was free from iron the product is white.]

POTASSIUM ARSENITE SOLUTION.

LIQUOR POTASSII ARSENITIS.

[Liquor Arsenicalis. Fowler's Solution.]

Arsenous oxide, in fine powder	10 Gm
Potassium bicarbonate	20 Gm
Compound tincture of lavender	30 ml
Distilled water, sufficient.	

Boil the arsenous oxide and potassium bicarbonate with 100 ml of distilled water until solution has been effected. Then add enough distilled water to make the solution, when cold, measure 970 ml, and, lastly, add the compound spirit of lavender. Filter through paper.

Reaction. $As_2O_3+4KHCO_3=2K_2HAsO_3+H_2O+4CO_2$.

Notes. Prior to the revision of 1890 the Pharmacopœia of the United States contained a working formula for this preparation which called for only one-half as much potassium bicarbonate as is necessary. The proportions were, in other words, equal parts of arsenous oxide and potassium bicarbonate. The complaints frequently made that arsenic separated from Fowler's solution on standing may have been due to that error. If 12 Gm of KOH be used instead of the 20 Gm of $KHCO_3$ the arsenous oxide dissolves much more rapidly and the final result is the same. It is necessary that the compound tincture of lavender be added last in order to obtain a clear solution.

Description.—A clear liquid, having a reddish color and an

aromatic odor derived from the compound tincture of lavender. It has an alkaline reaction on litmus paper.

POTASSIUM BENZOATE.

POTASSII BENZOAS.

$KC_7H_5O_2.3H_2O=214.$

Potassium bicarbonate	82 parts
Benzoic acid	100 parts
Distilled water	500 parts

Dissolve, neutralize, filter and evaporate to dryness, or to crystallization.

White, odorless, or of faint benzoin odor; sweetish; readily soluble.

POTASSIUM BICARBONATE.

POTASSII BICARBONAS.

$KHCO_3=100.$

Prepared by treating potassium carbonate with carbonic acid gas. The gas is either conducted into a concentrated solution of the carbonate, or over the moist salt. To facilitate the absorption of carbonic acid the potash is mixed with powdered charcoal. The moist mass is exposed to the action of the carbonic acid gas until a test portion in solution no longer precipitates a solution of magnesium sulphate.

The black mass is mixed with water, heated to about 70° or 80° C., the solution is filtered while warm to remove the charcoal and the silica which has been precipitated by the carbonic acid, and the filtrate is set aside in a cool place to crystallize. The crystals are carefully washed to remove adhering mother liquor, which contains normal carbonate, a little bicarbonate, chloride, and sulphate of potassium.

Description.—A white salt, crystalline, and soluble in 3.2 parts of water. Insoluble in alcohol. When absolutely pure the reaction is neutral or only very slightly alkaline. By heat it is converted into the monocarbonate with loss of CO_2. Dissolved in

water it loses carbonic acid even at ordinary temperatures, but rapidly and to complete conversion into normal carbonate above 50° C. In the dry state it does not readily part with any of its carbonic acid until at 100° C.

POTASSIUM BITARTRATE.

POTASSII BITARTRAS.

$KHC_4H_4O_6 = 188.$

In the manufacture of wine a large amount of impure potassium bitartrate is deposited from the grape juice during the second fermentation, the salt being thrown out of solution by the alcohol formed. Thick crusts are formed in the vats, over the bottom and sides. The bitartrate formed on the bottom contains more calcium salt than that on the sides. The "argols" or "crude tartar" must be powdered, redissolved in boiling water, freed from coloring matter by the addition of clay, which carries it down in settling. The solution is then clarified with albumen, strained, and allowed to deposit crystals. The crystals forming on the sides are again less impure than those on the bottom. Their relative freedom from calcium salt is indicated by their clearness or opacity, the milky opaque crystals being more contaminated with calcium tartrate.

This purified argols or refined tartar is further purified by reducing it to fine powder and digesting this with pure hydrochloric acid diluted with distilled water. The calcium tartrate is then decomposed by the hydrochloric acid, forming calcium chloride and free tartaric acid, both of which are washed out. The mixture is now allowed to cool, so that most of the potassium bitartrate dissolved in the water may deposit again, after which the "cream of tartar" is washed with cold water until the washings cease to give any reaction for chlorides, and then dried. A small percentage of bitartrate is lost by solution in the water with which it is treated.

Description.—Colorless or slightly opaque crystals or a white powder having no odor, but an agreeable acidulous taste. Soluble in 201 parts of water at 15°; but in 16.7 parts of boiling water.

BORAX TARTAR.

TARTARUS BORAXATUS.

[Boro-Tartrate of Potassium and Sodium.]

Borax, in powder	2 parts
Potassium bitartrate	5 parts
Boiling water	20 parts

Put the salts in the water, contained in a porcelain dish, and stir until all is dissolved. Filter. Evaporate the solution until a thread of the semi-liquid residue becomes brittle on cooling. Then pull it into sticks, or flatten it out in thin cakes, and dry it by the aid of heat. Powder the product in a warm mortar, and keep it in tightly closed bottles.

Notes. In some pharmacopœias the proportions are 1 part of borax to 2 parts of cream of tartar. There is undoubtedly a chemical union of some kind between the two salts, but the product is not a definite chemical compound. However, when 1 part of borax and 3 parts of potassium bitartrate are combined, a double salt is formed having the composition (according to Duve):

$$KNaC_4H_3(BO)O_6+KHC_4H_3(BO)O_6.$$

It is sometimes directed that the cream of tartar should be added gradually to the borax previously dissolved in the water. This it not only unnecessary, but delays the operation considerably. If both salts are added at once the solution of both is far more rapid than the solution of borax alone would be. Constant stirring is, however, necessary, because otherwise the borax on the bottom of the dish cakes together and afterwards dissolves rather slowly.

When large amounts are operated upon, water-bath heat is insufficient for evaporating the solution to dryness; steam-heat and constant stirring must be employed. Having evaporated the solution to dryness we have a yellowish gummy residue, which on cooling has a glassy fracture. This is then dried in thin cakes or threads at 70° C.; it is sufficiently dry when white and porous.

To powder the product fine is both difficult and disadvantageous; it should only be reduced to a very coarse powder, in a warm mortar, and then at once put into warm, dry bottles which must be tightly corked.

Description.—A coarse, white powder, dry, very deliquescent, soluble in its own weight of cold water, but insoluble in alcohol. It has a pleasant acidulous taste.

Potassium Boro-Tartrate.

Potassium bitartrate	4 parts
Boric acid	1 part
Boiling water	10 parts

Heat together with constant stirring until dissolved. Filter, and evaporate to dryness.

Powder the residue.

Description.—A white powder having an agreeable acidulous taste. It is not deliquescent. Soluble in twice its weight of water; insoluble in alcohol.

POTASSIUM BROMATE.

POTASSII BROMAS.

$KBrO_3$=167.

Bromine	87 parts
Potassium hydroxide	61 parts
Water	100 parts

Dissolve the potassium hydroxide in the water in a half-liter flask. Add the bromine very gradually, keeping the flask in *cold* water and shaking well after each addition of bromine. The solution, after all of the bromine has been added, should be neutral and colorless. If yellow or red add more potassium hydroxide until slightly alkaline and let stand until colorless, after which neutralize carefully with hydrobromic acid. Filter the liquid and set it aside in a cold place for some hours. Collect the crystals of bromate which have separated, drain them well. Redissolve the salt in three times its weight of boiling distilled water,

filter the solution, and set it in a cool place. When cold, put it in an ice-water bath. Collect, drain and dry the crystals.

The residuary solutions contain potassium bromide which should be recovered in the usual way.

Reaction. $6KOH+3Br_2=5KBr+KBrO_3+3H_2O$.

Description.—White or colorless crystals. Odorless. Taste saline. Soluble in 14 parts of water at 20°; in about 3 parts at 80°, and in about 2 parts of boiling water.

POTASSIUM BROMIDE.

POTASSII BROMIDUM.

KBr=119.

Iron wire, cut	5 parts
Bromine	12 parts
Potassium bicarbonate	15 parts
Distilled water.	

Put the iron together with 40 parts of distilled water in a flask. Add 9 parts of bromine, in small portions at a time, shaking the flask after each addition. Heat the flask and contents if necessary to cause the reaction to be completed—i. e., until all odor of bromine ceases and a green solution of ferrous bromide results. Filter the solution. Add the remainder of the bromine to the filtrate and shake well.

Put the potassium bicarbonate in a porcelain dish with 50 parts of distilled water, heat until effervescence ceases, and then raise the heat to boiling.

Add slowly to the boiling solution of potassium carbonate the solution of bromide of iron. Boil the mixture for fifteen minutes. The solution should now have a slightly alkaline reaction. If not, add more potassium carbonate or bicarbonate until the reaction is alkaline.

Let the precipitate subside. Filter the solution. Wash the precipitate on the filter with hot distilled water and add the washings to the filtrate. Evaporate the solution to the requisite degree of concentration and set it aside to crystallize. Drain the crys-

tals in a funnel. Then spread the salt upon bibulous paper and dry it with the aid of moderate heat.

Additional crystals may be obtained from the mother-liquor in the usual way.

Instead of crystallizing the salt the solution may be evaporated to dryness and the bromide granulated.

Reaction.

$Fe+2Br=FeBr_2$; then $3FeBr_2+Br_2=FeBr_2.2FeBr_3$;

and, finally, $FeBr_2.2FeBr_3+4K_2CO_3$

$=8KBr+FeO.Fe_2O_3+4CO_2$.

Notes. Ferroso-ferric bromide gives better results than ferrous bromide because the iron is more readily and completely precipitated in the form of ferroso-feric oxide.

Potassium bromide crystallizes best from a slightly alkaline solution. But if the salt is to be recovered in a granular condition by evaporation to dryness with constant stirring, then the solution should be neutral. It is, however, necessary to render the solution faintly alkaline for the purpose of precipitating all the iron. The liquid can afterwards be made neutral again by adding enough hydrobromic acid, which, for this purpose, may well be made from potassium bromide and tartaric acid.

To improve the appearance of potassium bromide the crystals may, after drying, be heated for a few minutes at a temperature of 80° to 100°.

Second Method.

Solution of potassium hydroxide.....	250 parts
Bromine, about.....	20 parts
Wood charcoal, in fine powder.....	10 parts
Boiling distilled water.....	150 parts

Put the solution of hydroxide in a flask; add the bromine gradually, with constant agitation, until the liquid has acquired a permanently brown color. Evaporate to dryness; powder the residue and mix it well with the charcoal. Throw the mixture into a red hot iron crucible, and when reduced to a state of fusion, pour out the contents, let cool, dissolve it in the boiling water,

filter, and set aside to crystallize. Drain the crystals and dry them in a warm place. Recover the salt contained in the mother liquor by evaporation to dryness, stirring constantly.

Reaction.

$3Br_2+6KOH=5KBr+KBrO_3+3H_2O$; and then,

$2KBrO_3+3C=2KBr+3CO_2$.

Notes. A stronger solution of potassium hydroxide than the official "liquor potassæ" (5%) may well be used. In that case the reaction must be controlled to prevent the liquid from becoming too hot; this may be accomplished by keeping the flask in cold water while the bromine is being very slowly added. See also notes under Potassium Iodide.

Third Method.

Ammonium bromide.....	98 parts
Potassium bicarbonate.....	100 parts

Dissolve the ammonium bromide in 100 parts of hot water; add the potassium bicarbonate; boil the solution until all odor of ammonia has ceased. Then crystallize the potassium bromide.

Description.—Colorless or white cubical crystals, or a white granular salt; odorless, and of saline, pungent taste. Soluble at 15° in about 1.6 parts of water and in 200 parts of alcohol; in less than its own weight of boiling water, and in 16 parts of boiling alcohol. Soluble in four times its weight of glycerin. The water-solution must have a neutral reaction on test-paper, or only a very faintly alkaline reaction.

POTASSIUM CARBONATE.

POTASSII CARBONAS.

[Potash. Pearl-Ash. Salts of Tartar.]

$K_2CO_3=138$.

Potassium carbonate is contained in wood ashes, and may be washed out of the ashes by lixiviation, the salt being recovered

from the solution by boiling it down to dryness. The residue is then purified by heating it strongly in a furnace until all organic matters have been destroyed. Calcined potash is the result; it contains 50 to 80 per cent of potassium carbonate, and is contaminated with sulphate, chloride, etc. By converting this crude potassium carbonate into bicarbonate, most of the impurities are removed, and a nearly pure normal carbonate can then be prepared from the bicarbonate.

Formerly, pure potassium carbonate was prepared by strongly heating (detonating) potassium bitartrate with half its weight of potassium nitrate.

At Stassfurth, potassium carbonate is prepared as follows: A solution of magnesium sulphate (*kieserite*) is mixed with a solution of crude potassium chloride, both of the materials being taken from the Stassfurth mines; a double salt of sulphate of potassium and magnesium is then formed together with a double chloride (*carnallite*): $3KCl+2MgSO_4=K_2Mg(SO_4)_2+KMgCl_3$. The double sulphate is boiled with more potassium chloride, which results in the formation of potassium sulphate and carnallite. The carnallite decomposes slowly in water so that the magnesium chloride can be separated, and the potassium chloride is then used over again as before—

$$K_2Mg(SO_4)_2+3KCl=2K_2SO_4+KCl+MgCl_2.$$

The potassium sulphate is then turned into carbonate by a process analogous to Leblanc's method for manufacturing soda. The resulting product contains 92 to 93 per cent of potassium carbonate, 2 or 3 per cent of sodium carbonate, 2 per cent of potassium chloride, and 1 to 2 per cent of potassium sulphate.

Crude potash may be partly purified by dissolving it in twice its weight of water, letting it stand two days, decanting, filtering through linen, and evaporating the clear liquid in a clean iron vessel until a pellicle begins to form on the surface. The liquid is then put in a stone pot and set aside for a week, in order that sulphates, chlorides, etc., may crystallize out. The solution is then filtered and boiled down to dryness in an iron pot, stirring constantly at the close so as to obtain a dry, granular product.

Pure potassium carbonate for pharmaceutical and medicinal uses is most readily obtained by heating potassium bicarbonate

until the residue is entirely soluble in twice its weight of water, or until it has lost from 30 to 31 per cent of its weight.

Being very deliquescent, potassium carbonate must be kept in tightly closed vessels.

Preparation of Potassium Carbonate from Bicarbonate.

Potassium bicarbonate, any desired quantity.

Put the salt into a porcelain dish, cover it well with distilled water, heat gradually and continue the heat until effervescence ceases and the liquid boils. Then evaporate to dryness, stirring constantly. Put the product at once into a perfectly dry bottle and close this tightly.

Reaction. $2KHCO_3=K_2CO_3+H_2O+CO_2$.

Notes. To obtain a perfect product the potassium bicarbonate used must be perfectly clean and pure. If it makes an unclear solution in water, the bicarbonate should all be dissolved in about five or six times its weight of water and the solution filtered before it is heated to decompose it into normal carbonate, water and carbon dioxide.

The product must be perfectly dry and white.

Description.—Potassium carbonate, sufficiently pure to fulfill the requirements of the Pharmacopœia, is a white granular powder, odorless, of strongly alkaline taste. It is very deliquescent. Soluble in 1.1 parts of water at 15°, and in 0.65 part of boiling water. Insoluble in alcohol.

POTASSIUM CHLORATE.

POTASSII CHLORAS.

$KClO_3=122.4$.

Formerly prepared by saturating a solution of potassium hydroxide or carbonate with chlorine, whereby hypochlorite and chloride of potassium were formed, the hypochlorite being afterwards decomposed by heat into chloride and chlorate:

$2KOH+Cl_2=KClO+KCl+H_2O$; and then,

$3KClO=KClO_3+2KCl$.

The chlorate being much less soluble than the chloride is readily separated by crystallization from the solution.

Liebig's method, now used, consists in treating a mixture of calcium hydroxide and potassium chloride with chlorine, when calcium chlorate is formed. Then the calcium chlorate and potassium chloride interact to form potassium chlorate and calcium chloride. The product is readily crystallized out from the liquid and purified by recrystallization.

Turbidated Potassium Chlorate.

$KClO_3$=122.4.

Potassium chlorate	1 part
Boiling distilled water	5 parts

Dissolve and filter. Stir the filtrate constantly until cold. Collect the crystals on a filter, let drain, and then dry the product.

Notes. Commercial potassium chlorate usually contains potassium chloride and calcium chloride. These impurities are much more readily water-soluble than potassium chlorate. Hence the turbidation of the salt results also in purification, for the chlorides remain in the mother liquor.

When cooled to 15° C. the mother liquor still retains about 6 per cent of its weight of chlorate, or 30 per cent of the amount of salt operated upon. This must be recovered as far as practicable, which can be effected by evaporating the mother-liquor by water-bath heat to about one-third its bulk, stirring until cold, and collecting, draining and drying the "second crop" of small crystals in the same manner as the first.

Description.—Potassium chlorate consists of colorless crystals, or a glistening, white, crystalline powder, odorless, of a cooling saline taste. Soluble in 16.7 parts of water at 15°; in about 3 parts of water at 75°, and in 1.7 parts of boiling water. Nearly insoluble in alcohol. Explosive with certain readily oxidized substances, especially when heated with them, or mixed with reducing agents by trituration, or by concussion. A powerful oxidizing agent.

POTASSIUM CHLORIDE.

POTASSII CHLORIDUM.

$KCl=74.4$.

Potassium bicarbonate	100 parts
Diluted hydrochloric acid	364 parts
Distilled water	100 parts

Heat the potassium bicarbonate in a porcelain dish with the water until converted into normal carbonate. Then add this gradually to the hydrochloric acid, stirring well. When the solution is neutral to test-paper, filter it, and evaporate the filtrate to crystallization or granulation, as may be desired.

Reaction. $K_2CO_3+2HCl=2KCl+H_2O+CO_2$.

Purification of crude potassium chloride. The commercial crude salt contains sulphates and chlorides of magnesium, sodium and calcium. It is purified as follows:

Crude chloride	2 parts
Water	5 parts

Dissolve the salt in the water with the aid of heat (about 80°). Add about 0.05 part of lime previously slaked and mixed with ten times its weight of water to precipitate magnesium. Stir well. Filter. Add to the filtrate enough solution of barium chloride to precipitate the sulphates. Filter again. Then add enough solution of ammonium carbonate to precipitate the calcium and barium. Filter a third time. Evaporate the filtrate to dryness and heat the residue at a low red heat to decompose the ammonium salt. Dissolve the mass in water, acidulate the solution with hydrochloric acid, boil the liquid a few minutes, filter, and evaporate to the density of 1.20. Set aside to crystallize. Drain and dry the crystals.

Description.—Colorless translucent crystals, or a white granular salt; odorless and of a pure saline taste. Soluble at 15° in about three times its weight of water, and in less than one-half its weight of boiling water. Insoluble in alcohol.

POTASSIUM CITRATE.

POTASSII CITRAS.

$K_3C_6H_5O_7.H_2O=324.$

Potassium bicarbonate	10 parts
Citric acid	7 parts

Crush the citric acid and the bicarbonate, separately; add the latter gradually to 50 parts of boiling water, and when effervescence ceases add the citric acid, a small portion at a time, until the liquid is neutral to litmus paper, or but faintly alkaline. Filter, and evaporate to dryness, stirring constantly after a pellicle has commenced to form, so that the product may be granular.

Reaction.

$H_3C_6H_5O_7+3KHCO_3=K_3C_6H_5O_7.H_2O+3CO_2+2H_2O;$

or, $2H_3C_6H_5O_7+3K_2CO_3=2K_3C_6H_5O_7+3CO_2+3H_2O.$

Notes. The temperature must not be too high toward the close of the evaporation, as citric acid and citrates are decomposed at a high heat.

The product should be perfectly white, and must be entirely dry before it is bottled.

Description.—White, transparent crystals, or a white granular powder; odorless, and of a pure saline, cooling taste. Hygroscopic. Soluble in 0.6 part of water at 15°, and freely soluble in boiling water. Sparingly soluble in alcohol. Neutral to litmus paper.

Potassium Citrate Solution.

LIQUOR POTASSII CITRATIS.

Citric acid	6 parts
Potassium bicarbonate	8 parts

Dissolve each in 40 parts of cold water. Filter the solutions separately, and wash the filters with enough water to obtain, in each case, 50 parts of filtrate. Mix the two solutions, and, when

effervescence has nearly ceased, put the product in a bottle and cork it tightly.

Should be freshly prepared whenever required for use.

Notes. It is intended that cold water should be used, and that the preparation should retain in solution as much of the carbonic acid as may be soluble in that volume of the watery liquid. An excess of citric acid is purposely employed in order that the preparation may have a slightly acidulous taste.

Description.—A clear, colorless, liquid, of pure mildly saline taste and slightly acid reaction.

Effervescent Potassium Citrate.

Citric acid	63 Gm
Potassium bicarbonate	90 Gm
Sugar	47 Gm

Powder the ingredients separately, and mix them thoroughly in a warm mortar. Dry the resulting, uniform paste rapidly at a temperature not exceeding 120° C., and, when it is perfectly dry, reduce it to a powder of the desired degree of fineness.

Keep the product in well-stoppered bottles.

Notes. A partial reaction ensues when the ingredients are triturated together in a warm (not *hot*) mortar, on account of the water always contained in the citric acid, and the liberation of a portion of water formed by the reaction renders the mixture somewhat pasty at the moderate heat employed. When the mixture is dissolved in water a complete reaction takes place and normal potassium citrate is formed:

$$3KHCO_3+H_3C_6H_5O_7.H_2O=K_3C_6H_5O_7.H_2O+3H_2O+3CO_2.$$

Potassium Boro-Citrate.

Citric acid	10 Gm
Boric acid	9 Gm
Potassium carbonate	15 Gm
Water	60 ml

Dissolve and evaporate to dryness, stirring constantly.

Description.—A perfectly white salt, readily forming a clear solution in water.

POTASSIUM CYANATE.

POTASSII CYANAS.

KCNO=81.

Anhydrous potassium ferrocyanide.....	4 parts
Dry potassium dichromate.....	3 parts
Alcohol	18 parts
Methyl alcohol.....	2 parts

Melt the potassium dichromate, and, while it is still warm, mix it with the powdered anhydrous potassium ferrocyanide. Put the mixture, a small spoonful at a time, into a large iron dish heated over a gas stove, stirring with an iron spatula after each addition so that each portion of powder added is converted into a black mass, taking care not to raise the temperature so high that the mass fuses.

Powder the black mass while still warm. Then put it in a flask and heat it in a water-bath for ten minutes with a mixture of the two alcohols, shaking the contents frequently and well.

Decant the clear solution through a pleated filter into a beaker placed in crushed ice.

Set the flask containing the undissolved black residue in ice water without delay.

After the crystals have deposited in the beaker, pour the mother liquor back over the black mass in the flask.

Repeat the extractions of the cyanate from the residue if necessary.

Drain the crystals, wash them with strong ether and dry them *in vacuo* over sulphuric acid.

Notes. To make anhydrous potassium ferrocyanide, heat the crushed commercial salt in an iron dish slowly until completely effloresced, or until no yellow unchanged salt remains in the interior of any piece. Then powder it while still warm and complete the drying by heating the powder spread out in a thin layer on a hot iron plate or dish for two or three hours.

When the mixed salts are heated to a black mass there should be no evolution of ammonia and there will be none if the materials were first thoroughly dried.

The yield is about 1.7 parts. The product is very unstable. Decomposed by water.

POTASSIUM CYANIDE.

POTASSII CYANIDUM.

KCy=65.

Potassium ferrocyanide, dried and powdered	8 parts
Potassium carbonate, dry	3 parts

Mix the powdered materials well, throw the mixture into a deep iron crucible previously heated to dull redness, and maintain the heat until effervescence ceases and the fused mass solidifies on cooling, which may be ascertained by dipping a warm glass rod in it and withdrawing. When the fused cyanide assumes a pure white color on cooling, pour it out into a shallow dish, and, when solid, break it into pieces and put it in dry bottles while still warm.

Reaction. $K_4FeCy_6+K_2CO_3=5KCy+KOCy+CO_2+Fe$.

Notes. Care is to be taken to decant the fused cyanide properly from the residue—that is, the pouring must be discontinued as soon as the decanted fused salt is no longer white.

The product contains cyanate as well as cyanide.

Another process consists in conducting hydrocyanic acid into solution of potassa to which alcohol has been added. The cyanide formed is then deposited in small crystals, which are to be drained on a funnel, washed with strong alcohol, in which the preparation is nearly insoluble, and dried between bibulous paper at a moderate heat.

Being deliquescent, and readily absorbing carbonic acid from the air with the formation and loss of hydrocyanic acid, the product must be kept in tightly closed bottles.

Description.—White, amorphous pieces, or a white, granular powder, odorless when dry, but in moist air emitting the odor of hydrocyanic acid. The taste is sharp, and somewhat alkaline.

Deliquescent in moist air. *Very poisonous.*

Soluble in about 2 parts of water at 15° C. Boiling water dissolves its own weight of the salt, but rapidly decomposes it. In alcohol it is but sparingly soluble.

POTASSIUM DICHROMATE.

POTASSII DICHROMAS.

$K_2Cr_2O_7$=294.

[Potassium Bichromate.]

Chromite, in powder	6 parts
Potassium carbonate	3 parts
Lime	8 parts
Potassium sulphate	1 part
Sulphuric acid.	
Water.	

Dissolve the potassium carbonate in 3 parts of water.

Slake the lime in a large porcelain dish with about two-thirds of the solution of potassium carbonate. Then add the remainder of that solution and mix the whole well. Evaporate the mixture to dryness. Heat the residue to 150° and stir it until reduced to powder. Mix this with the powdered chromite.

Heat the dry mixture to a bright red heat for three or four hours in free contact with air, stirring frequently.

Let the mass cool, powder it, and mix the powder with 20 parts of water, stirring well. Strain the solution and evaporate the colature until crystals begin to form.

Dissolve the potassium sulphate in 1 part of boiling water, and add this solution gradually to the concentrated solution of chromate, so long as precipitation of calcium sulphate is produced by it. Then filter while hot.

To the hot yellow filtrate add sulphuric acid diluted with twice its volume of water until the liquid is strongly acid. Let the liquid cool. Collect the crystalline precipitate on an asbestos or glass wool filter, or on a bed of broken glass in a funnel. Purify the salt by recrystallization.

Drain the crystals of dichromate and dry them with the aid of moderate heat.

Reaction. $4Cr_2FeO_4+8K_2CO_3+7O_2=8K_2CrO_4+2Fe_2O_3+8CO_2$; and $4Cr_2FeO_4+8Ca(OH)_2+7O_2=8CaCrO_4+2Fe_2O_3+8H_2O$; then $CaCrO_4+K_2SO_4=K_2CrO_4+CaSO_4$; and, finally, $2K_2CrO_4+H_2SO_4=K_2Cr_2O_7+K_2SO_4+H_2O$.

Notes. The chromite consists mainly of Cr_2FeO_4 but also contains silicates. When ignited with the potash and lime it forms ferric oxide, the chromates of potassium and calcium, and potassium silicate. The ignition may be performed in a muffle furnace, or, more advantageously, in a reverbatory furnace, using the oxidation flame.

The lime is used in large excess to keep the mass porous when ignited, and a portion of it forms chromate so that the amount of potassium carbonate required is lessened. The chromates are leeched out of the mass with water, and the potassium sulphate is added to convert the calcium chromate into potassium chromate, calcium sulphate being precipitated. The liquid containing the chromates is yellow.

When sulphuric acid is added to the solution of potassium chromate the color of the liquid is at once changed from yellow to orange red, and, as the potassium dichromate now formed is much less soluble in water than the chromate, a large proportion of the dichromate crystallizes out on cooling. The quantity of water prescribed is adjusted with this view.

The first crop thus obtained is nearly free from potassium sulphate and may be purified by one recrystallization.

The mother liquor can be concentrated by evaporation and more crystals obtained, but the second and subsequent crops of dichromate are difficult to purify because they are so largely contaminated with potassium sulphate.

Beautiful crystals of potassium dichromate can be easily obtained by recrystallizing it from a solution of 1 part of the salt in 10 parts of water, allowing the filtered solution to evaporate spontaneously.

Turbidated potassium dichromate may be conveniently prepared from a solution of 1 part of the salt in 5 parts of boiling water.

The mother liquors obtained in recrystallizing and in turbidating the dichromate yield all of the salt they contain, on evaporation to dryness.

Description.—Large, clear, transparent, orange-red crystals; odorless; taste, bitter, metallic. Soluble in 10 parts of water at 15°, and in 1.5 parts of boiling water. Insoluble in alcohol.

The water-solution turns blue litmus paper red.

POTASSIUM CHROMATE.

POTASSII CHROMAS.

K_2CrO_4=194.5.

Potassium dichromate.....10 parts
Potassium carbonate..... 5 parts
Water.

Put the potassium dichromate in a porcelain dish with 30 parts of water. Heat to about 80°. Add the potassium carbonate, in small portions at a time, stirring well. When all of the carbonate has been added heat the liquid to boiling, add 20 parts of water, filter, and evaporate to crystallization. Drain and dry the crystals with the aid of moderate heat.

Recover the remainder of the salt from the mother liquor in the usual way.

Reaction. $K_2Cr_2O_7+K_2CO_3=2K_2CrO_4+CO_2$.

Description.—Yellow translucent crystals. Soluble in about 1.5 parts of water at 15°.

POTASSIUM FERRICYANIDE.

POTASSII FERRICYANIDUM.

$K_6Fe(CN)_{12}$=658.

Potassium ferrocyanide.....100 parts
Lead dioxide.
Water.

Dissolve the ferrocyanide in 150 parts of water, with the aid of heat, in a flask capable of holding about 600 parts of water. Pass a current of carbon dioxide into the solution heated to the boiling point. Add 10 parts of lead dioxide to the hot solution, and stir well. Boil for an hour or longer, passing a stream of carbon dioxide into the liquid during that time.

Should the precipitated lead carbonate cause too violent bumping remove it by filtering the hot liquid through a muslin filter,

add a fresh portion of 10 parts of lead dioxide, boil again, and continue passing carbon dioxide into the liquid.

Repeat this treatment with lead dioxide and carbon dioxide until the filtered solution no longer gives a blue precipitate with solution of ferric chloride. Then let the solution cool in a covered vessel. Decant the clear liquid from any crystals which may have separated. Collect the crystals. Evaporate the solution over a water-bath until crystals begin to be formed on the surface. Then add a small quantity of hot water to redissolve the crystals, and filter the liquid while hot to separate the precipitate formed during the evaporation. Set the filtrate aside in a covered vessel to cool and crystallize as before. Collect the second crop of crystals and add it to the first. Drain and dry the crystallized product.

Reaction. $4K_4Fe(CN)_6+2PbO_2+4CO_2+2H_2O=4KHCO_3+2PbO+2K_6Fe_2(CN)_{12}$.

Notes. Several crops of crystals may be collected from the successive mother-liquors on their evaporation. Should yellow crystals of ferrocyanide begin to be formed, repeat the boiling with lead dioxide and treatment with carbon dioxide. The recovery of ferricyanide must be discontinued as soon as the product becomes contaminated with potassium bicarbonate.

Recrystallization may be necessary to render the product, or later crops of it, clean and pure.

Description.—Clear, transparent, blood-red crystals, freely soluble in water, forming a greenish-yellow solution.

POTASSIUM FERROCYANIDE.

POTASSII FERROCYANIDUM.

(Yellow Prussiate of Potash.)

$K_4Fe(CN)_6.3H_2O=422$.

Large, transparent, pale yellow, soft, odorless crystals, having a saline taste. Slightly efflorescent. Soluble in 4 parts of water at 15°, and in 2 parts of boiling water. Insoluble in alcohol.

Recrystallized Potassium Ferrocyanide.

Yellow prussiate of potash.....1 part
Water .....4 parts

Dissolve with the aid of heat. Filter. Crystallize at rest by spontaneous evaporation.

Very large crystals can be obtained. Turn the crystals now and then to permit their development in all directions.

Description.—Large, soft, transparent, light-yellow crystals, odorless, of mild saline taste. Soluble in 4 parts of water at 15°, and in 2 parts of boiling water. Insoluble in alcohol. The water-solution is neutral to litmus paper.

POTASSIUM HYDROXIDE.

POTASSII HYDROXIDUM.

("Potassa.")

KOH=56.

A strong solution of potassium hydroxide, prepared as described below, may be evaporated to dryness, and the residue purified with alcohol.

When impure potassium hydroxide is dissolved in alcohol, any carbonate, sulphate, and the greater portion of any chloride present remain undissolved, and may thus be removed. The alcohol may then be distilled off from the clear, decanted solution, and the remaining mass is dried and fused in a silver capsule. Although the solution is brown from the action of the alkali upon the organic substances in the alcohol, the fused potassium hydroxide will still be white. To prevent the formation of carbonate, a little boiled distilled water is added from time to time to the alcoholic solution during its evaporation.

Notes. Potassium hydroxide must be kept in bottles of hard glass, as it attacks soft glass. Green glass free from lead is best. Glass stoppers in bottles containing potassium hydroxide or its solution, are acted upon by the alkali, and often become so firmly fastened to the neck that they cannot be removed. To prevent this a little petrolatum may be rubbed upon the stopper before inserting

it. This also effectually excludes air, and thus protects the potassium hydroxide from moisture and carbonic acid.

Description.—Dry, white, translucent, hard, brittle, odorless, very acrid, caustic, corrosive, rapidly deliquescent when exposed. Soluble at 15° in half its weight of water, and in twice its weight of alcohol. Freely soluble in boiling water and in boiling alcohol. Intensely alkaline.

Potassium Hydroxide Solution.

LIQUOR POTASSII HYDROXIDI.

Solution of Potassa.

An aqueous solution containing about 5 per cent. of potassium hydroxide (KOH, 56.)

Potassium bicarbonate	90 Gm.
Lime	40 Gm.
Distilled water, sufficient.	

Dissolve the potassium bicarbonate in 400 ml of distilled water, heat the solution until effervescence ceases, and then raise it to boiling. Slake the lime and make it into a smooth mixture with 400 ml of distilled water, and heat it to boiling. Then gradually add the first liquid to the second and continue the boiling for ten minutes. Remove the heat, cover the vessel tightly, and, when the contents are cold, add enough distilled water to make the whole weigh 1,000 Gm. Lastly, strain through linen, or remove the clear solution, after subsidence of the precipitate, by means of a syphon.

Solution of potassium hydroxide should be kept in well stoppered bottles.

Reaction. The potassium bicarbonate is first converted into normal carbonate by boiling the solution. Then, $K_2CO_3 + Ca(OH)_2 = 2KOH + CaCO_3$.

Notes. The potassium bicarbonate is used in preference to monocarbonate, because bicarbonate is readily obtained nearly pure, whereas carbonate usually contains various impurities. The lime is used in considerable excess, but no lime will be contained

Recrystallized Potassium Ferrocyanide.

Yellow prussiate of potash.....1 part
Water .....4 parts

Dissolve with the aid of heat. Filter. Crystallize at rest by spontaneous evaporation.

Very large crystals can be obtained. Turn the crystals now and then to permit their development in all directions.

Description.—Large, soft, transparent, light-yellow crystals, odorless, of mild saline taste. Soluble in 4 parts of water at 15°, and in 2 parts of boiling water. Insoluble in alcohol. The water-solution is neutral to litmus paper.

POTASSIUM HYDROXIDE.

POTASSII HYDROXIDUM.

("Potassa.")

KOH=56.

A strong solution of potassium hydroxide, prepared as described below, may be evaporated to dryness, and the residue purified with alcohol.

When impure potassium hydroxide is dissolved in alcohol, any carbonate, sulphate, and the greater portion of any chloride present remain undissolved, and may thus be removed. The alcohol may then be distilled off from the clear, decanted solution, and the remaining mass is dried and fused in a silver capsule. Although the solution is brown from the action of the alkali upon the organic substances in the alcohol, the fused potassium hydroxide will still be white. To prevent the formation of carbonate, a little boiled distilled water is added from time to time to the alcoholic solution during its evaporation.

Notes. Potassium hydroxide must be kept in bottles of hard glass, as it attacks soft glass. Green glass free from lead is best. Glass stoppers in bottles containing potassium hydroxide or its solution, are acted upon by the alkali, and often become so firmly fastened to the neck that they cannot be removed. To prevent this a little petrolatum may be rubbed upon the stopper before inserting

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An aqueous solution containing about 5 per cent. of potassium hydroxide (KOH, 56.)

Potassium bicarbonate	90 Gm.
Lime	40 Gm.
Distilled water, sufficient.	

Dissolve the potassium bicarbonate in 400 ml of distilled water, heat the solution until effervescence ceases, and then raise it to boiling. Slake the lime and make it into a smooth mixture with 400 ml of distilled water, and heat it to boiling. Then gradually add the first liquid to the second and continue the boiling for ten minutes. Remove the heat, cover the vessel tightly, and, when the contents are cold, add enough distilled water to make the whole weigh 1,000 Gm. Lastly, strain through linen, or remove the clear solution, after subsidence of the precipitate, by means of a syphon.

Solution of potassium hydroxide should be kept in well stoppered bottles.

Reaction. The potassium bicarbonate is first converted into normal carbonate by boiling the solution. Then, $K_2CO_3 + Ca(OH)_2 = 2KOH + CaCO_3$.

Notes. The potassium bicarbonate is used in preference to monocarbonate, because bicarbonate is readily obtained nearly pure, whereas carbonate usually contains various impurities. The lime is used in considerable excess, but no lime will be contained

in the finished solution of potassium hydroxide, as calcium hydroxide is insoluble in that liquid. Theoretically 1 part of lime is sufficient to causticize 3.6 parts of potassium bicarbonate when the process is conducted as prescribed in the official formula, which is the one given above. It would be a decided improvement upon this process to first wash the calcium hydroxide, throwing away the first portion of water used as in the process for making lime water; this removes dust, salts, etc. An unnecessarily large excess of lime is disadvantageous in that the calcium hydroxide settles more slowly than the carbonate.

Potassium carbonate may be causticized by calcium hydroxide without the aid of heat, but the calcium carbonate then formed is very light, settles slowly, and retains much liquid. By using hot solutions, as directed by the Pharmacopœia, a much more dense calcium carbonate is formed which settles rapidly, so that the solution can be separated with less waste.

Not less than 8 parts, and preferably 12 parts of water should be taken for each part of normal potassium carbonate used. The process of the U. S. Pharmacopœia employs about 10½ parts, for 90 parts of bicarbonate corresponds to about 77 parts of carbonate, and the water used is 800 parts. If a more concentrated solution is used the decomposition will be incomplete. The reaction is complete when the liquid no longer effervesces with dilute acid, and does not cause a turbidness when dropped into clear lime water.

Solution of potassium hydroxide can well be evaporated in a clean, bright iron pot down to about 1.16 specific weight without danger of attacking the iron. A stronger solution, however, would attack the metal. In any event a solution of potassium hydroxide should not be allowed to remain long in contact with iron, and whenever such a solution is to be concentrated in an iron vessel, the evaporation should be hastened as much as possible.

Straining or filtering the solution is liable to cause its discoloration. It is, therefore, best to let it stand long enough to become clear by subsidence and then to draw off the clear liquid by means of a glass syphon.

The glass-stoppered bottles used to contain it should be made of green glass free from lead, and the stoppers rubbed with petrolatum.

Instead of directing that 1,000 Gm. of the solution should be made out of 90 Gm. of potassium bicarbonate, thus making the strength of the product depend upon the amount of material taken and upon the care exercised in the management of the details of the process, the Pharmacopœia ought to direct that the strength be determined by actual test with volumetric solution of sulphuric acid *before* it is finished instead of afterwards, and the preparation adjusted, by dilution with distilled water, to the prescribed standard; in other words, the quantitative test should be a part of the process of preparation.

Description.—Clear, colorless, odorless, acrid, caustic, strongly alkaline. Sp. w. about 1.036 at 15°.

Valuation. To neutralize 28 Gm. of the official solution of potassium hydroxide (5%) requires 25 ml of normal sulphuric acid. Each ml of the volumetric sulphuric acid solution indicates 0.2 per cent. of KOH. Phenolphtalein is the indicator used. One ml of normal sulphuric acid is the equivalent of 0.05599 Gm. of KOH.

Potassa with Lime.

POTASSA CUM CALCE.

Potassa	1 part
Lime	1 part

Rub them together, in a warm iron mortar, so as to form a powder, and keep it in a well-stoppered bottle.

Notes. The lime prevents the potassium hydroxide from absorbing carbon dioxide from the air to form carbonate. The mixture is, however, less caustic and corrosive than potassium hydroxide alone. The glass stopper of the bottle in which the preparation is kept should be coated with petrolatum.

Description.—A grayish-white powder, deliquescent, having a strongly alkaline reaction, and responding to the tests for calcium and potassium. It should be soluble in diluted hydrochloric acid without leaving more than a small residue.

POTASSIUM HYPOPHOSPHITE.

POTASSII HYPOPHOSPHIS.

$KPO_2H_2 = 104$.

Calcium hypophosphite	1 part
Potassium carbonate	1 part

Dissolve each salt separately in 7 parts of boiling water. Mix the solutions. Filter off the calcium carbonate, and evaporate the filtrate to dryness, over a water-bath, stirring constantly so as to obtain a granular product.

Notes. The temperature during the evaporation should not exceed 85° C., as hypophosphites are prone to decompose with violent explosion when heated to nearly 100° C., or higher.

The product may be freed from potassium carbonate and other impurities by dissolving it in ten times its weight of alcohol, filtering, evaporating the solution to the consistence of thick syrup, and setting this aside to solidify into a crystalline mass.

Reaction. $Ca(PO_2H_2)_2 + K_2CO_3 = CaCO_3 + 2KPO_2H_2$.

Description.—A white granular salt, easily soluble in water and weak alcohol. It may be obtained in crystalline form from the aqueous solution.

POTASSIUM HYPOTHIOSULPHITE.

POTASSA SULPHURATA.

Sulphurated Potassa.

(Liver of Sulphur.)

Sublimed sulphur	1 part
Potassium carbonate	2 parts

Mix by trituration. Heat the mixture gradually in a covered crucible, which should be only half-filled, until the mass ceases to swell and is completely melted. Then pour the molten mass upon a cold stone or glass slab, and, when it has become hard

and nearly cold, break it into pieces and keep it in a dry bottle of hard glass which must be tightly closed.

Reaction. When the proportions of the materials are as here given, the reaction is frequently represented as $4K_2CO_3+10S=3K_2SS_2+K_2SO_4+4CO_2$; but the following is probably the principal reaction: $3K_2CO_3+8S_2=K_2S_2O_3+2K_2SS_2+3CO_2$. The composition of the product depends much upon the temperature, which should not be too high. It is quite probable that sulphur is soluble in fused potassium sulphide (K_2S) as well as in a water solution of it. The proportion of sulphur used is in some formulas greater and in others less; yet the product is in each case physically homogeneous. When a solution of sulphurated potassa, U. S. P., is mixed with a solution of copper sulphate the reaction between them is: $CuSO_4+K_2S_3=CuS+K_2SO_4+S_2$. From the fact that only one-third of the sulphur contained in the sulphurated potassa forms CuS in this case, it is obvious that the sulphur is not all of one kind. In other words the sulphur in sulphurated potassa made by fusing sulphur and potassium carbonate together is partly positive and partly negative, as would be the case in K_2SS_2.

Notes. The potassium carbonate and the sulphur must both be quite dry. They should be finely powdered and well mixed in order to obtain a uniform product. It is well to pass the powdered mixture through a sieve of about 600 meshes to the square centimeter.

A covered iron dish will answer well for the fusion, and a moderate heat (from 150° to 250° C.) should be used. The mass should be occasionally stirred.

The heat should not be higher than is necessary to fusion without turning the mixture into a thin liquid.

When the evolution of carbon dioxide ceases, and the whole mass fuses quietly and completely, a sample should be taken out and put into about twice its weight of water; when it dissolves entirely, *without separation of sulphur*, the fused mass is allowed to cool somewhat, and is then poured upon a slab (or into an oiled iron dish), and covered well (with a bell glass or an inverted dish) to exclude air and moisture, until nearly cold. It is then broken and immediately transferred to a dry container.

Description.—Liver brown pieces having a bitter alkaline taste

and an odor of hydrogen sulphide. Hygroscopic; soluble in twice its weight of water at 15° C., leaving a small residue. Alcohol dissolves the potassium sulphide, leaving the sulphate and thiosulphate.

POTASSIUM IODATE.

POTASSII IODAS.

$KIO_3 = 213.5$.

Potassium permanganate..... 2 parts
Potassium iodide..... 1 part
Water,
Alcohol,
Acetic Acid.

Dissolve the permanganate in 50 parts of hot water, add the iodide dissolved in 2 parts of water, heat the mixture over a water-bath for about twenty minutes or half an hour, adding alcohol drop by drop at the end until the liquid is decolorized, and then filter. Wash the precipitated potassium maganite on the filter and add the washings to the filtrate. To the filtrate add enough acetic acid to render the reaction on test-paper distinctly acid. Evaporate the liquid to about 1.5 parts; let it cool; separate the crystals from the mother-liquor, wash the product with alcohol and dry it.

Reaction. $KI + 2KMnO_4 = KIO_3 + K_2Mn_2O_5$.

Notes. The acidification with acetic acid is necessary to prevent the product from having an alkaline reaction due to adhering alkali, which it is extremely difficult to wash away.

Description.—A white, granular, crystalline salt of perfectly neutral reaction to test-paper.

POTASSIUM IODIDE.

POTASSI IODIDUM.

$KI = 165.5$.

Solution of potassium hydroxide.....1,000 ml
Iodine .....100 Gm., or sufficient
Wood charcoal, in fine powder..... 15 Gm
Boiling distilled water, sufficient.

Put the solution of potassium hydroxide in a flask, add the iodine, a little at a time, with constant agitation, until the solution becomes permanently brown. Evaporate to dryness. Powder the residue, and mix it well with the charcoal. Throw the mixture into a red-hot iron crucible, and when the whole is in a fused condition pour it out to cool, dissolve it in 200 ml of boiling distilled water, filter, and evaporate until a pellicle forms. Then set aside to cool and crystallize. Drain the crystals and dry them in a warm place. The remainder of the salt may be obtained from the mother liquor by evaporation to dryness, stirring constantly.

Reaction. $3I_2+6KOH=5KI+KIO_3+3H_2O$; and then,

$2KIO_3+3C=2KI+3CO_2$.

Notes. The red brown liquid formed by dissolving iodine in solution of potassium hydroxide contains potassium iodide and potassium iodate, as seen from the above equation. By heating the salt mass obtained on evaporation of this liquid to dryness, the iodate is reduced to iodide; but the heat necessary for this purpose is so high that loss of iodine ensues, and the product becomes alkaline. To prevent this, charcoal is added, which makes the reduction practicable at a much lower temperature.

The crystallization of potassium iodide is somewhat difficult. It is best to continue the evaporation until a sample of the liquid, when removed from the vessel, crystallizes on cooling. The liquid should then be left in a warm place (50° to 60°), where the evaporation may continue spontaneously in order that large crystals may be obtained. At a lower temperature the iodide ascends the sides of the vessel and creeps over the edges.

When perfectly pure, potassium iodide is difficult to obtain in colorless crystals; it undergoes partial decomposition with liberation of enough iodine to discolor the product. To prevent this discoloration the liquid is usually rendered slightly alkaline before being set aside for crystallization. The presence of potassium carbonate not only facilitates the formation of crystals, but also renders the product whiter. The pharmacopœial tests permit a slight alkalinity (less than 0.1 per cent). Crystals of perfectly pure potassium iodide are usually not quite white, but may be made so by drying at 120° to 125° C.

Second Method.

Iron wire, cut	6 parts
Iodine	20 parts
Potassium bicarbonate	16 parts
Distilled water, sufficient.	

Digest the iron and 15 parts of the iodine with 50 parts of water in a flask until all odor of iodine ceases and a green solution of ferrous iodide results. Filter this, and in the filtrate dissolve the remainder of the iodine.

Put the potassium bicarbonate into a dish with 80 parts of distilled water, heat until effervescence ceases, and then raise the heat to boiling. Add slowly to this boiling hot solution the filtered solution of the iodides or iron. Boil the mixture for fifteen minutes. (If all the iron has not been precipitated from the solution, add a little more potassium bicarbonate.) Filter, evaporate, and crystallize.

Reaction. $Fe+I_2=FeI_2$; then $3FeI_2+I_2=FeI_2.2FeI_3$; finally

$$FeI_2.2FeI_3+4K_2CO_3=8KI+FeO.Fe_2O_3+4CO_2.$$

Notes. As seen from the equation, a solution of ferrous iodide is first made. This can be at once treated with potassium carbonate to obtain the potassium iodide:

$$FeI_2+K_2CO_3=2KI+FeCO_3.$$

But in this case the iron is separated with greater difficulty, being first thrown down as ferrous carbonate, which at once begins to lose carbonic acid, and at the same time oxidizes so that the precipitate gradually changes from light gray to blue, yellowish brown, and finally to brown ferric hydroxide $Fe(OH)_3$. All of the iron does not separate at once, however, so that during the subsequent evaporation of the filtered solution of iodide of potassium a considerable quantity of flocculent deposit of ferric hydroxide makes its appearance and must be filtered out.

This difficulty may be remedied to a great extent by converting the ferrous iodide into ferrico-ferrous iodide by adding the required additional quantity of iodine. The precipitate will then, after the boiling, be black magnetic oxide of iron, which is very dense and, therefore, more easily separated than ferric hydroxide.

When the solution of iodide of iron has all been added to the hot solution of potassium carbonate, the liquid should have a neutral or faintly alkaline reaction. Should it instead be acid, add more potassium bicarbonate. If too alkaline add some hydriodic acid prepared from tartaric acid and KI.

Third Method.

Red phosphorus	1 part
Iodine	12 parts
Potassium carbonate	6 parts
Distilled water, sufficient.	

Put the phosphorus in a porcelain dish with 35 parts of distilled water and warm the liquid over a water-bath to about 40° C. Add the iodine a little at a time, stirring gently, until the liquid becomes permanently colored.

Decant the clear solution from the residue, wash the latter with a little distilled water, and add the washings to the decanted solution.

Add enough calcium hydroxide mixed with water ("milk of lime") to impart a slightly alkaline reaction to the liquid.

Filter the solution, and add the potassium carbonate dissolved in 10 parts of water, stirring well.

Decant the liquid from the precipitate, wash the latter with some distilled water, add the washings to the decanted solution, and filter the liquid.

Evaporate the solution to crystallization.

Reactions. The first reactions result in the formation of PI_3 and PI_5. Then: $PI_3+3H_2O=H_3PO_3+3HI$; and

$H_3PO_3+I_2+H_2O=H_3PO_4+2HI$. At the same time this reaction occurs: $PI_5+4H_2O=H_3PO_4+5HI$. Hence two molecules of H_3PO_4 and ten molecules of HI are the final products.

When calcium hydroxide is added the reactions are:

$2HI+Ca(OH)_2=CaI_2+2H_2O$ and

$H_3PO_4+Ca(OH)_2=CaHPO_4+2H_2O$ and

$2CaHPO_4+Ca(OH)_2=Ca_3(PO_4)_2+2H_2O$.

When the calcium phosphate has been filtered out and the potassium carbonate is added to the solution of CaI_2 the reaction is:

$$CaI_2+K_2CO_3=2KI+CaCO_3.$$

Notes. The use of red instead of ordinary phosphorus lessens the danger involved in this process. But great care should be exercised lest the phosphorus iodides decompose with violence. After the addition of the prescribed amount of potassium carbonate to the solution of calcium iodide, the filtered liquid should be tested with a few drops of potassium carbonate solution, and, if further precipitation is caused thereby, more potassium carbonate solution should be cautiously added, a little at a time, stirring well, until precipitation is no longer produced by it.

The final solution, before evaporation, should be of a slightly alkaline reaction. Instead of evaporating to crystallization the salt may be granulated.

Description.—Colorless or white translucent cubical crystals or a granular crystalline powder, with a faint iodine-like odor and pungent, saline, bitterish taste. Slightly hygroscopic. Soluble at 15° in 0.75 part of water and in 18 parts of alcohol; in 0.5 part of boiling water, and in 6 parts of boiling alcohol. The water-solution has a neutral or only faintly alkaline reaction on test-paper.

POTASSIUM NITRATE.

POTASSII NITRAS.

$KNO_3=101.$

Potassium chloride..... 6 parts
Sodium nitrate..... 7 parts
Water.

Mix the salts in a porcelain dish with 12 parts of water. Heat at the boiling point, stirring well and without interruption for several minutes. Filter. Evaporate the filtrate to 8 parts. Should any salt separate during the evaporation heat the liquid to boiling and filter again. Let the filtrate stand several hours. Collect the crystals of potassium nitrate and recrystallize several times to remove adhering sodium chloride. Dry the product perfectly.

Reaction. $KCl+NaNO_3=KNO_3+NaCl$.

Notes. No reaction takes place until the solution is boiled down. But the process is based upon the differences in the respective solubilities of sodium chloride and potassium nitrate at different temperatures. Potassium nitrate is less readily soluble in cold water than the chlorides of potassium and sodium, but far more freely soluble than the chlorides in hot water. Thus the sodium chloride crystallizes out from the hot saturated solution of potassium nitrate. The separation of the sodium chloride is facilitated by having the sodium nitrate in excess; but the product should be washed with a cold saturated solution of potassium nitrate to remove the last traces of chlorides and sodium nitrate.

Commercial potassium nitrate may be purified by dissolving it in an equal weight of boiling distilled water, filtering, and stirring the filtrate constantly until cool. The saltpeter should first be coarsely powdered to facilitate its solution.

When cool, set it aside for some hours. Collect the crystals on a filter, and let drain. Wash twice by rapidly passing small quantities of distilled water over the crystals, allowing all of the wash-water to pass through before adding more. When the washings no longer give a decided reaction for chlorides (with test solution of silver nitrate), collect the crystals and dry them between blotting paper, or on suitable trays covered with muslin.

Evaporate the mother liquor at about 95° C. to one-third its bulk, and let cool during constant stirring. Wash the second crop of crystals as before, drain, and dry as before, and add them to the first crop. Repeat this operation as often as may be profitable.

To simply granulate potassium nitrate it may be dissolved in an equal weight of boiling distilled water, the solution filtered, and the filtrate evaporated, stirring constantly with a glass rod or porcelain spatula until a dry, granular salt remains.

Description.—Colorless crystals, or a crystalline granular powder, odorless, and of a cooling, saline, pungent taste. Soluble in 3.8 parts of water at 15°, and in 0.4 part of boiling water. Very sparingly soluble in alcohol. Alcohol of 80% strength dissolves less than one-half of one per cent of its own weight of potassium nitrate; 100 parts of alcohol of 50% strength dissolves only 2.8

parts of the salt, and 100 parts of alcohol of 10% strength 13.2 parts.

Turbidated Potassium Nitrate.

Potassium nitrate	1 part
Boiling water	2 parts

Dissolve, filter, and stir until cold. Collect, drain, and dry the crystals.

Evaporate the mother-liquor nearly to dryness, stirring constantly, and dry the residue, which should be collected separately, or turbidated as before if the quantity is sufficient to render it advantageous.

POTASSIUM OLEATE.

Soft Soap.

[Sapo Mollis. Green Soap. Potash Soap.]

Linseed oil	400 Gm
Potassium hydroxide (90%)	90 Gm
Alcohol	40 ml
Water, sufficient.	

Heat the oil in a deep porcelain dish on a water-bath to a temperature of about 60° C. Dissolve the potassium hydroxide in 450 ml of water, add the alcohol to the solution, and then gradually add this mixture to the oil, stirring constantly, continuing the heat and stirring until the product becomes perfectly soluble in boiling water without the separation of oily drops (which may be ascertained by tasting a small portion from time to time). Then allow the product to cool and transfer it to suitable containers.

Description.—A soft, brownish-yellow solid, translucent in thin layers. Should make an almost clear solution in 5 parts of water. Should dissolve in 2 parts of hot alcohol, leaving not over 3 per cent of undissolved residue.

POTASSIUM OXALATE; NORMAL.

POTASSII OXALAS.

$K_2C_2O_4.2H_2O=202.$

Oxalic acid	63 parts
Potassium bicarbonate	100 parts
Distilled water	500 parts

Dissolve the oxalic acid in the water by the aid of heat; then add the potassium bicarbonate. Neutralize perfectly. Evaporate and crystallize.

Reaction. $2KHCO_3+H_2C_2O_4=K_2C_2O_4+2H_2O+2CO_2.$

Description.—Transparent prisms or pyramids, easily soluble in water.

ACID POTASSIUM OXALATE.

$KHC_2O_4.2H_2O=162.$

Oxalic acid	126 Gm
Potassium bicarbonate	100 Gm
Distilled water	1,000 ml

Dissolve the potassium bicarbonate in the water by the aid of heat, boiling the liquid until effervescence ceases; then add the oxalic acid, dissolve, filter while hot, and set aside to cool and crystallize.

Reactions. $2KHCO_3=K_2CO_3+H_2O+CO_2$; then,

$K_2CO_3+2H_2C_2O_4=2KHC_2O_4+H_2O+CO_2.$

Description.—Occurs usually as anhydrous prisms or crystallized with one or two molecules of water.

It is not readily soluble in cold water.

POTASSIUM PERMANGANATE.

POTASSII PERMANGANAS.

$KMnO_4$=158.

Manganese dioxide	60 parts
Potassium hydroxide	70 parts
Potassium chlorate	35 parts
Water.	

Heat the finely powdered manganese dioxide to a red heat. Let it cool again. Put the potassium hydroxide in a porcelain dish, add 100 parts of water, and then the potassium chlorate. Heat until solution is effected. Then add the manganese dioxide. Evaporate the mixture to a thick pasty mass, stirring constantly during the evaporation to make the mixture uniform. Heat the paste to redness in an iron crucible or dish, which should not be more than half filled as the mass swells on heating. When water vapors cease to escape and the mass becomes dark green or brown and quite hard, let it cool, remove it from the crucible or dish, and powder it. Mix it with about 350 parts of water, boil the mixture for an hour, passing a current of carbon dioxide into the boiling liquid during that period. The green color of the liquid is changed to violet by this treatment and a brown precipitate is thrown down. Set the liquid aside until the precipitate has subsided. Filter the solution through asbestos or glass wool. Wash the precipitate with a small quantity of hot water, and add the washings to the other filtrate. Evaporate rapidly until crystals begin to separate. Then allow the solution to cool in a well covered vessel. Collect the crystals. Evaporate the mother-liquor, after adding a little hydrochloric or sulphuric acid to it, and again let crystals be formed. Recrystallize this second crop of crystals before adding it to the first. Drain the crystals by suction and dry them over sulphuric acid.

Reactions.

$3MnO_2+6KOH+KClO_3=3K_2MnO_4+KCl+3H_2O$; then,

$3K_2MnO_4+2H_2O=2KMnO_4+MnO_2+4KOH$; and,

$KOH+CO_2=KHCO_3$.

Description.—Slender, opaque, dark purple, lustrous crystals, odorless; taste sweetish, disagreeable, astringent. Soluble in 16 parts of water at 15°, and in 3 parts of boiling water. Decomposed in contact with alcohol. Explosive with glycerine.

POTASSIUM PHOSPHATE.

POTASSII PHOSPHAS.

$K_2HPO_4 = 174$.

Prepared in the same manner as sodium phosphate, using potassium carbonate to decompose the acid phosphate of calcium.

Potassium phosphate is a deliquescent, indistinctly crystalline, white mass, very freely soluble in water. At red heat it is converted into pyrophosphate of potassium, which is also deliquescent.

POTASSIUM SALICYLATE.

POTASSII SALICYLAS.

$2KC_7H_5O_3.H_2O = 194$.

Potassium bicarbonate	29 parts
Salicylic acid	41 parts
Distilled water	150 parts

Dissolve, filter, evaporate to dryness.

White, odorless, acrid; freely soluble

POTASSIUM-SODIUM TARTRATE.

POTASSII ET SODII TARTRAS.

Rochelle Salt.

[The British Pharmacopœia calls Rochelle Salt "Tartarated Soda."]

$KNaC_4H_4O_6.4H_2O = 282$.

Potassium bitartrate	5 parts
Sodium carbonate	4 parts
Distilled water	20 parts

Heat the water to boiling in a porcelain dish; add the sodium carbonate, and when that is dissolved, gradually add the cream

of tartar, and continue heating until all is dissolved. Set the solution aside for a day or two. Then filter, evaporate the filtrate to nine parts, or until a pellicle forms, and then set it aside to crystallize.

Reaction.

$$2KHC_4H_4O_6+Na_2CO_3=2KNaC_4H_4O_6+H_2O+CO_2.$$

Notes. A slight excess of sodium carbonate is prescribed, because the salt crystallizes best from a slightly alkaline solution. Any excess of sodium carbonate will remain in the mother liquor. Large crystals are easily obtained. The crystals should be hastily rinsed with a little cold distilled water. The mother liquor is usually colored, more so the farther the evaporation is carried. (See, also, Potassium Tartrate.)

The direction to allow the solution to stand some time before filtering and evaporating to crystallization, has for its object the deposition of any calcium tartrate and carbonate.

The crystals must be dried without the aid of heat.

After several crystallizations, the mother-liquor becomes colored. It may be decolorized with animal charcoal if the quantity operated upon justifies it. The tartaric acid in the last mother-liquor may be recovered in the form of cream of tartar by precipitation with hydrochloric acid.

Description.—Colorless, transparent prisms, or a white powder; odorless; taste cooling, saline. Slightly efflorescent in warm, dry air. Soluble in 1.4 parts of water at 15°, and in less than its own weight of boiling water. Practically insoluble in alcohol. Neutral to litmus paper.

POTASSIUM THIOCYANATE.

POTASSII THIOCYANAS.

(Potassium Sulphocyanate. Rhodankalium.)

KCNS=97.

Anhydrous potassium ferrocyanide.....	46 parts
Potassium carbonate.....	17 parts
Washed sulphur.....	32 parts
Alcohol.	

Mix the thoroughly dried and powdered solids very intimately by triturating them together in a mortar. Heat the mixture slowly until the mass fuses. Let it cool. Extract the thiocyanate with hot alcohol and collect the crystals which deposit on cooling. Evaporate the mother-liquor to obtain more.

Notes. For directions how to make the anhydrous potassium ferrocyanide see notes under potassium cyanate.

Description.—A white crystalline salt, of bitter saline taste. Readily water-soluble.

POTASSIUM SULPHATE.

POTASSII SULPHAS.

$K_2SO_4=174$.

Diluted sulphuric acid.....	100 parts
Potassium bicarbonate.....	20 parts

Place the acid in a porcelain dish and neutralize it with the potassium bicarbonate, gradually added, stirring after each addition until effervescence has ceased before adding more. Try the solution with test-paper, and add more potassium bicarbonate if necessary to render the reaction quite neutral. Filter the solution and evaporate to crystallization.

Reaction. $2KHCO_3+H_2SO_4=K_2SO_4+2H_2O+2CO_2$.

Description.—Colorless, odorless crystals of a somewhat bitterish saline taste. Soluble in 9.5 parts of water at 15°, and in 4 parts of boiling water. Insoluble in alcohol.

POTASSIUM SULPHITE.

POTASSII SULPHIS.

$K_2SO_3.2H_2O=194$.

Prepared by passing sulphurous oxide into a solution of potassium carbonate until all carbonic acid has been expelled, and then adding another equal quantity of potassium carbonate. The

solution is then evaporated to crystallization. See Sodium Sulphite.

Description.—Colorless crystals or powder, readily soluble in water, and but slightly in alcohol. Decomposed by acids yielding SO_2 without deposit of sulphur.

POTASSIUM TARTRATE.

POTASSII TARTRAS.

$K_2C_4H_4O_6.H_2O=470.$

Potassium bicarbonate..... 15 parts
Cream of tartar..... 28 parts
Distilled water.

Dissolve the potassium bicarbonate in 50 parts of water with the aid of sufficient heat to cause effervescence. Raise the temperature of the liquid to the boiling point. Add the cream of tartar in small portions at a time, stirring well until all has dissolved, leaving the liquid faintly alkaline in its reaction on test-paper. Filter. Evaporate the filtrate until signs of crystallization appear. Then set the dish aside for three or four days in a cold place. Collect the crystals in a funnel and let them be well drained.

Evaporate the mother liquor over a water-bath to one-third of its volume and again set aside to crystallize.

Dry the crystals by moderate heat.

Keep the product in a tightly closed bottle.

Reaction.

$$K_2CO_3+2KHC_4H_4O_6=2K_2C_4H_4O_6.H_2O+CO_2.$$

Notes. Should the reaction of the solution not be faintly alkaline, make it so by the addition of more of the carbonate or the tartrate as may be required. The salt crystallizes best from a slightly alkaline solution.

When colorless crystals can no longer be obtained from the mother-liquor, dilute the liquid with an equal volume of water, filter, and add hydrochloric acid as long as any precipitate is

formed. This precipitate is acid tartrate of potassium, which should be collected, washed, and dried.

Description.—Clear colorless crystals, soluble in 0.75 part of water at 15°, and in 0.50 part of boiling water. Odorless; taste saline, mildly bitterish. It is slightly hygroscopic.

SILVER; PURE.

ARGENTUM PURUM.

Ag=108.

Pure or sterling silver may be obtained from silversmiths. But coined silver, and the silver used in the manufacture of silverware is mixed with copper to harden it. This alloy can be purified as follows:

The silver coin or old silver is dissolved in nitric acid as described under the title of Argenti Nitras; the solution is diluted with distilled water and filtered. A filtered hot solution of sodium chloride is then added to it as long as it causes precipitation. The precipitated silver chloride is washed with boiling distilled water and then dried. The dried chloride is thoroughly mixed with an equal weight of dry potassium carbonate, and the mixture is heated in a clay crucible, first at a low red heat for about half an hour, or until the silver chloride has been decomposed, and without causing the mass to fuse; after which the heat is increased until the whole contents of the crucible undergoes complete fusion so that the metallic silver runs together forming a button or lump.

Crystallized Silver.

Silver..... 60 parts
Nitric acid (68% HNO_3)..... 70 parts
Water.
Stronger ammonia water.
Sodium bisulphite.

Dissolve the silver in the acid previously diluted with 100 parts of water, with the aid of gentle heat, in the fume chamber. Evaporate the solution to dryness. Heat the residue in a por-

celain dish until it fuses. Continue heating until the fused salt *begins* to turn black. Let it cool. Dissolve the mass in 150 parts of water and filter the solution. Add ammonia water, stirring well, until the liquid acquires a decided ammoniacal odor.

Make a forty per cent solution of sodium bisulphite in water and filter. Add a sufficient quantity of this solution to the ammoniacal silver solution to reduce the cupric salt in the latter to the cuprous state. This may be known to have been accomplished when a sample of the blue liquid becomes decolorized on heating it.

Let the liquid stand in a cold place until most of the silver has crystallized out, which it does slowly. Collect the crystals, wash them with cold distilled water and then cover them with strong ammonia water and let stand for a few hours. Wash again with distilled water and then dry.

To recover the remainder of the silver from the mother liquor heat it at from 60° to 70° until all of the metal has precipitated, and wash and dry this precipitate in the same way as the other.

SILVER CHLORIDE.

ARGENTI CHLORIDUM.

$AgCl=143.4$.

Silver nitrate	17 parts
Sodium chloride	6 parts
Distilled water.	

Dissolve the silver nitrate in 200 parts of distilled water, and the sodium chloride in 100 parts. Filter both solutions. Heat the sodium chloride solution to boiling. Add the silver solution to that of the sodium chloride, stirring well. Wash the precipitated silver chloride with boiling distilled water until the washings are perfectly tasteless. Dry the product with the aid of moderate heat.

Keep it in dark amber-colored bottles.

Reaction. $AgNO_3+NaCl=AgCl+NaNO_3$.

Description.—A white, heavy, insoluble, odorless and tasteless powder. Readily soluble in ammonia water. Darkened by light.

SILVER CYANIDE.

ARGENTI CYANIDUM.

AgCy=134.

Silver nitrate	34 parts
Potassium cyanide	13 parts
Distilled water.	

Dissolve the silver nitrate in 500 parts and the potassium cyanide in 200 parts of distilled water. Add the solution of potassium cyanide to the solution of silver nitrate, gradually, and stir well. Wash the precipitate with distilled water, and dry it with the aid of moderate heat. Keep it well protected against light.

Reaction. $AgNO_3+KCy=AgCy+KNO_3$.

Notes. The solution of silver nitrate might advantageously be acidulated with nitric acid.

Description.—A heavy, white, odorless and tasteless powder. Insoluble in water or alcohol. Darkens on exposure to light.

SILVER IODIDE.

ARGENTI IODIDUM.

AgI=234.5.

Silver nitrate	1 part
Potassium iodide	1 part

Dissolve the salts separately, each in about 12 parts of distilled water. Pour the solution of silver nitrate gradually and with constant stirring into the solution of the iodide. Collect the precipitate on a filter, wash it well with distilled water, and dry it between bibulous paper.

The product is to be kept in well closed bottles and protected from light.

Reaction. $AgNO_3+KI=AgI+KNO_3$.

Description.—A heavy, light-yellowish powder; odorless and tasteless. Insoluble in water and alcohol.

SILVER NITRATE.

ARGENTI NITRAS.

$AgNO_3 = 170$.

Silver	30 parts
Nitric acid (containing 68% of HNO_3)	35 parts
Distilled water	50 parts

Add the nitric acid and the water to the metal in a flask, and heat gently until dissolved. Decant the clear liquid into a porcelain capsule, evaporate, and set aside to crystallize. Let the crystals drain in a glass funnel, and dry them by exposure to the hot air, carefully avoiding contact with organic matter.

Keep it in an amber colored bottle, with glass stopper.

Reaction. $3Ag + 4HNO_3 = 3AgNO_3 + 2H_2O + NO$.

Notes. If coin is used it should be well cleaned with hot soda solution before it is dissolved in the acid.

The metal dissolves rapidly at first, and with considerable evolution of heat. Afterwards, however, the reaction is slower as the solution becomes more concentrated. To prevent loss by spurting, the neck of the flask may be covered by a watch-crystal.

When large quantities are operated upon the flask may be set in a warm place and left for several days. The clear liquid is then decanted from the residue, and the latter is washed, the filtered washings being added to the other solution. It is best to have the metal present in excess so that a small quantity of it remains undissolved. By this precaution vapors of nitric acid are avoided in the subsequent evaporation.

To remove copper, if present, the solution is evaporated to dryness and the mass fused in a thin porcelain capsule (Meissen dish) over a sand-bath, keeping the salt fused two or three hours until a small quantity of it, when removed from the dish, dissolved in water, and the solution filtered, is not turned blue by ammonia water. All of the copper nitrate which was present has now been reduced to cupric oxide. The mass is then poured into a clean polished iron dish or mortar, and allowed to cool; it cannot be allowed to cool in the porcelain dish for that will burst.

The salt is then redissolved in distilled water, the solution is filtered, and crystallization effected.

Both fused and crystallized silver nitrate are free from water.

In evaporating a solution of silver nitrate to crystallization sand-bath heat may advantageously be employed until crystals begin to form. Then a little nitric acid should be added, the dish should be transferred to a water-bath and evaporation continued until a sample of the solution solidifies on cooling. The dish is then put in a dark place and well covered. The crystals are to be drained in a funnel, and then dried with the aid of moderate heat.

The mother liquor should be evaporated to dryness to recover all of the silver nitrate.

Description.—Colorless, transparent, tabular crystals; odorless; taste bitter, caustic, nauseous, strongly metallic. Darkens on exposure to light in the presence of organic matter, especially if damp. Soluble at 15° in 0.6 part of water, and in 0.1 part of boiling water. Soluble in 26 parts of alcohol but is decomposed in alcoholic solution.

Fused Silver Nitrate. Lunar Caustic. Lapis Infernalis.)

Silver nitrate.....	100 parts
Hydrochloric acid.....	4 parts

Fuse the silver nitrate in a porcelain capsule on a sand-bath at as low a temperature as possible; then add the acid gradually, stirring well, and when nitrous vapors cease to be evolved, mould the salt into sticks or cones by means of a suitable mould of bright polished iron, or of silver.

Notes. By the addition of hydrochloric acid, silver chloride is introduced into the preparation: $AgNO_3+HCl=AgCl+HNO_3$. The object of this is to render the fused sticks more tough and strong.

Silver nitrate fused with 5 per cent of potassium chloride is also a useful toughened caustic containing enough silver chloride and potassium nitrate to be used in the usual sticks or pencils with lessened danger of breakage.

Description.—A heavy opaque solid, usually of a grayish color; fracture granular crystalline.

About 95 per cent of the mass is readily soluble in water. The remaining 5 per cent, consisting of chloride, is not. It is not so quickly soluble as the pure salt.

Diluted Silver Nitrate.

Silver nitrate	1 part
Potassium nitrate	2 parts

Fuse the salts together in a porcelain dish over a sand-bath at as low a temperature as possible, stirring constantly until the mass flows quietly. Then cast the melted mass in suitable moulds.

Notes. Silver nitrate alone, when fused and moulded into pencils, is too brittle, and it happens frequently that a less energetic caustic than the pure silver salt is desired. Hence the pharmacopœias prescribe mixtures of silver nitrate and potassium nitrate, and some pharmacopœias contain two such mixtures—one consisting of equal parts, and another of one part silver nitrate and two parts of potassium nitrate. The Pharmacopœia of the United States prescribes equal parts in the Sixth Revision (1880), but orders the more diluted kind in the Seventh Revision (1890).

Description.—A hard solid, finely crystalline, and readily soluble in water. It has a metallic taste and caustic properties similar to the pure nitrate.

Mixtures of silver nitrate and potassium nitrate in other proportions are also offered by manufacturers of chemicals, and distinguished from each other by numbers.

Silver Nitrate with Lead.

This caustic in pencils is made by fusing together 15 parts of lead nitrate and 85 parts of silver nitrate, moulding the mixture into sticks of the usual form. It is recommended as preferable to other silver nitrate caustics on account of its greater toughness and softness. It can be sharpened to a point with the knife, like a lead pencil.

SILVER OLEATE.

ARGENTI OLEAS.

$AgC_{18}H_{33}O_2=389.$

Crystallized silver nitrate	25 Gm
White castile soap, in fine powder	45 Gm

Dissolve the silver nitrate in 1,500 ml of cold distilled water, and the soap in 500 ml of hot water. Add the soap solution very slowly and with brisk stirring to the silver solution. Collect the oleate, drain it, and wash it several times with cold distilled water. Dry it without the aid of heat, protecting it from exposure to light or dust.

Reaction. $AgNO_3+NaC_{18}H_{33}O_2=AgC_{18}H_{33}O_2+NaNO_3.$

Notes. Silver oleate is very sensitive to light, heat, and contact with dust, impure air, etc. When first formed it is nearly white, but cannot be kept so unless it is prepared at night or in a dark room. Decomposition may be to a great extent prevented by surrounding and covering with yellow paper the funnel in which the oleate is washed. If properly precipitated it is finely granular, and it should be dried by the use of white blotting paper, frequently changed. If carefully protected the product will be of a light purplish white color.

The product is about 56 Gm, containing 29.77 per cent of silver oxide. It should be kept in small amber glass stoppered bottles, wrapped in yellow paper.

SILVER OXIDE.

ARGENTI OXIDUM.

$Ag_2O=232.$

Silver nitrate	2 parts
Distilled water	40 parts
Solution of potassium hydroxide (5%)	13 parts

Dissolve the silver nitrate in the water. To this solution add solution of potassa so long as any precipitate is produced by it.

Wash the precipitate with distilled water until the washings are nearly tasteless. Dry the product and keep it well protected from the light.

Reaction. $2AgNO_3+2KOH=Ag_2O+2KNO_3+H_2O$.

Notes. Instead of potassium hydroxide a corresponding amount of calcium hydroxide may be used. The proportions required are then 1 part of silver nitrate dissolved in 20 parts of water, and 135 parts of saturated solution of calcium hydroxide (lime water).

Silver oxide easily parts with its oxygen, and hence must not be brought in contact with readily oxidizable substances, as explosion may result. Moderate heat also decomposes it. With ammonia it forms a violently explosive compound known as "fulminate of silver." Great care is accordingly necessary in handling this preparation. It should be kept in a well-closed bottle and in a cool place.

Description.—A heavy, dark-brown, nearly black, powder; odorless; taste metallic. Nearly insoluble in water to which it, nevertheless, imparts an alkaline reaction.

SODIUM ACETATE.

SODII ACETAS.

$NaC_2H_3O_2.3H_2O=136$.

Lead acetate	20 parts
Sodium carbonate	15 parts
Acetic acid, sufficient.	

Dissolve the salts, each in sixty parts of water; filter, and add the solution of lead acetate to that of the sodium carbonate, stirring briskly. Set aside to settle. Decant the supernatant liquid; wash the precipitate with a little cold distilled water and add the washings to the decanted solution. Filter. Add enough acetic acid to the filtrate to render it distinctly acid to litmus paper; then evaporate it to 25 parts, and set it aside to cool and crystallize. Concentrate the mother liquor by evaporation, and crystallize again.

Must be kept in tightly corked bottles.

Reaction.

$$3(Pb(C_2H_3O_2)_2.3H_2O)+3(Na_2CO_3.10H_2O,)=$$

$$2PbCO_3.Pb(OH)_2+6(NaC_2H_3O_2.3H_2O)+20H_2O+CO_2.$$

The salt effloresces on exposure, and in order to safely dry the crystals it is important that the temperature should not exceed 30° C. (86° F.). Still better is it to dry the crystallized salt in a strong current of cold air produced by a blower.

Bye-product.—The precipitated lead carbonate should be further washed until the washings cease to give an alkaline reaction to test-paper, and should then be dried.

Another Method.

Acetic acid (36%)	100 parts
Sodium carbonate	85 parts
Water	100 parts

Put the acetic acid and water in a porcelain dish. Add the coarsely powdered or crushed sodium carbonate in small portions, stirring after each addition until effervescence ceases before adding another portion. When all of the sodium carbonate has been added heat the liquid to the boiling point. Then add

Sodium carbonate	2 or 3 parts

as may be required to render the solution quite neutral to test-paper, or only faintly acid. Filter. Evaporate the filtrate until a test-portion when cooled gives crystals. Then let the liquid stand until cold. Collect, drain, and dry the crystals.

Reaction.

$$Na_2CO_3+2HC_2H_3O_2=2NaC_2H_3O_2+H_2O+CO_2.$$

Notes. When the solution is to be tested as to its reaction on test-paper the portion taken for this test should be first diluted with about three times its volume of water. Should the liquid be found to have an alkaline reaction more acetic acid must be added (and the whole well mixed) until the reaction is slightly acid.

The crystals must be dried at the ordinary temperature.

When acetic acid containing empyreumatic products is used,

their odor comes out on neutralizing the acid. The acetate should then be carefully heated to complete fusion at a temperature of about 300° (and not exceeding 310°) for half an hour when the empyreumatic substances will be decomposed. The salt is then recrystallized.

Description.—Colorless, transparent crystals; odorless; taste saline, cooling. Efflorescent in warm dry air. Soluble in 1.4 parts of water and in 30 parts of alcohol at 15°; in half its weight of boiling water, and in twice its weight of boiling alcohol. The salt becomes liquid at 60°, and anhydrous at 123°. It decomposes at 315° C. Its water-solution (5%) turns red litmus paper blue, but should not redden phenolphtalein paper. If it reddens phenolphtalein it probably contains sodium carbonate.

SODIUM ARSENATE.

SODII ARSENAS.

$Na_2HAsO_4.7H_2O=312.$

Arsenous oxide	20 parts
Sodium nitrate	17 parts
Dried sodium carbonate	11 parts

Powder these substances and mix them intimately by trituration. Heat the mixture in a large covered clay crucible at a red heat until effervescence has ceased, and complete fusion takes place. Let the fused mass solidify on a porcelain surface, and, while still warm, put it into 85 parts of boiling distilled water, and stir until dissolved. Filter while hot, and set aside to crystallize. Collect, drain, and dry the crystals, and keep them in a tightly corked bottle.

Reaction.

$As_2O_3+2NaNO_3+Na_2CO_3=Na_4As_2O_7+N_2O_3+CO_2$; and,

$Na_4As_2O_7+15H_2O=2(Na_2HAsO_4.7H_2O)$.

Notes. The residue after the process of heating the mixture is sodium pyroarsenate, which is converted into orthoarsenate by water.

When crystallized at a low temperature sodium arsenate has 12 molecules of water of crystallization; at ordinary temperature it usually crystallizes with $7H_2O$; in dry air it effloresces, losing $5H_2O$; the last of the water is driven off at about 149° C., and when heated to redness it becomes converted into pyroarsenate, which, however, when dissolved in water becomes immediately converted back into arsenate.

Description.—Colorless, transparent, odorless crystals, of mild alkaline taste (very poisonous). Hygroscopic in moist air. Soluble in 4 parts of water at 15°, and freely soluble in boiling water. Very sparingly soluble in cold alcohol; soluble in 60 parts of boiling alcohol.

Solution of Sodium Arsenate; U. S.

Sodium arsenate, dried at 149° C.....	1 part
Distilled water.....	99 parts

Notes. Owing to the variable amount of water of crystallization found in the sodium arsenate of commerce, the Pharmacopœia directs that the salt should be heated at 149° C. (300° F.) until deprived of all its water of crystallization before it is weighed out, and used in making the official solution. See preceding paragraph.

SODIUM BENZOATE.

SODII BENZOAS.

$NaC_7H_5O_2$=144.

Benzoic acid.....	40 parts
Sodium bicarbonate.....	28 parts
Boiling water.....	80 parts

Mix the bezoic acid with the hot water, stir well, neutralize perfectly by adding the sodium bicarbonate, being careful to observe the reaction on litmus paper after effervescence has ceased. Filter. Evaporate to 55 parts; remove it from the source of heat and stir until cold. During the evaporation it is necessary to scrape down the benzoate from the sides of the capsule.

Reaction. $2HC_7H_5O_2+Na_2CO_3=2NaC_7H_5O_2+H_2O+CO_2$.

Description.—A white, semi-crystalline or amorphous powder, efflorescent on exposure to air, odorless or having a faint odor of benzoin, of a sweetly astringent taste free from bitterness, and having a neutral reaction. Soluble in 1.8 parts of water, and in 45 parts of alcohol at 15° C.; in 1.3 parts of boiling water, and in 20 parts of boiling alcohol.

SODIUM BICARBONATE.

SODII BICARBONAS.

$NaHCO_3$=84.

Prepared by saturating a solution of sodium carbonate with carbonic acid gas, or by conducting carbonic acid gas for several days into closed chambers filled with crystallized sodium carbonate. The liberated water of crystallization retains in solution the chloride and sulphate which may have been present in the carbonate.

It may also be prepared as follows:

Stronger ammonia water (28% of H_3N)	250 Gm
Sodium chloride	250 Gm
Water,	
Carbon dioxide.	

Put the ammonia solution in a flask of 2000 Cc. capacity. Add one liter of water. Pass carbon dioxide into the mixture until saturated, keeping the flask cool by means of a stream of cold water.

Dissolve the sodium chloride in 800 Gm of water at 15°. Add to this the cold solution of ammonium bicarbonate, stirring well, and continuing the addition so long as a precipitate is formed. When no further precipitation results from the addition of more of the solution of ammonium bicarbonate, collect the precipitated sodium bicarbonate, wash it with some cold water on a funnel, and dry it.

Reactions. $H_3N+H_2O+CO_2=H_4NHCO_3$; then $H_4NHCO_3+NaCl=NaHCO_3+H_4NCl$.

Notes. Ammonium bicarbonate is not freely soluble in water.

Should any separation of this salt occur while carbon dioxide is led into the ammonia water, a little more water must be added at the close of that part of the process to redissolve the ammonium bicarbonate thus precipitated, before the liquid is added to the solution of sodium chloride.

Purification. Sodium bicarbonate, as found in commerce, is frequently impure from chlorides and sulphates. These impurities may be removed by washing with cold distilled water.

Close the throat of a glass funnel loosely with cotton. Fill the funnel about two-thirds full of the dry powdered commercial sodium bicarbonate. Cover the leveled surface of the salt with a disc of filter paper. Now pour upon the covered salt small quantities of cold distilled water, about 30 to 50 Gm at a time, and let the water percolate through the salt. After a quantity of water equal to rather more than half the weight of the sodium bicarbonate has passed through, test the washings for chlorides and sulphates in the usual way. When the washings, acidified with nitric acid, no longer become turbid on the addition of test-solution of either silver nitrate or barium chloride, dry the washed salt with the aid of very moderate heat.

Description.—A white, odorless powder, of cooling, mildly alkaline taste. Soluble in 11.3 parts of water at 15°. Above that temperature the salt begins to lose carbon dioxide, and at the boiling point of water it is converted into normal carbonate almost completely. At a low red heat it is completely converted into normal carbonate.

Insoluble in alcohol and in ether.

Vichy Salt.

Sodium bicarbonate	85 parts
Sodium chloride	5 parts
Sodium sulphate	9 parts
Potassium carbonate	1 part

Mix the separately powdered dry ingredients.

SODIUM BISULPHITE.

SODII BISULPHIS.

$NaHSO_3$=104.

Prepared by saturating a solution of sodium carbonate with sulphur dioxide and evaporating the solution to dryness.

Must be kept in small, entirely filled, tightly-closed bottles, in a cool place.

Description.—Opaque crystals or a white, granular powder, emitting a sulphurous odor; taste disagreeable, sulphurous. Oxidized by contact with air to sulphate, giving off sulphur dioxide. Soluble at 15° in 4 parts of water and in 72 parts of alcohol; in about 2 parts of boiling water and in 49 parts of boiling alcohol. Its water-solution exhibits an acid reaction on litmus paper.

SODIUM BITARTRATE.

SODII BITARTRAS.

$NaHC_4H_4O_6.H_2O$=190.

Tartaric acid	20 parts
Sodium carbonate	19 parts

Dissolve one-half of the acid in 50 parts of distilled water, neutralize with the sodium carbonate and filter. Dissolve the other half of the tartaric acid in 50 parts of distilled water and filter. Mix the two liquids. Set the mixture aside in a cool place for a day or two; then collect and dry the precipitated crystalline salt.

Description.—A white, crystalline powder, or colorless crystals; odorless; acid taste. Sparingly soluble in cold water. Insoluble in alcohol.

SODIUM BROMIDE.

SODII BROMIDUM.

NaBr=103.

Iron wire, cut	8 parts
Bromine	24 parts
Sodium carbonate	45 parts
Distilled water, sufficient.	

Digest the iron and 18 parts of bromine with 50 parts of water in a flask until all odor of bromine has ceased, and the liquid becomes green. Filter, add the remainder of the bromine to the filtrate, and let it dissolve.

Dissolve the sodium carbonate in 100 parts of boiling distilled water, and add the solution of bromide of iron. Boil for fifteen minutes, filter, and evaporate the filtrate to dryness during constant stirring.

Notes. The reactions are analogous to those occurring in the preparation of potassium bromide from bromide of iron and potassium carbonate.

The sodium bromide may also be made from bromine and solution of sodium hydroxide, as potassium bromide is prepared from bromine and solution of potassium hydroxide.

See also notes under Potassium Bromide.

Where the salt is deposited from hot concentrated solutions the crystals are anhydrous and cubical, while hydrated monoclinic crystals are deposited by slow evaporation of cold solutions.

Description.—Colorless or white crystals, or a white granular salt; odorless; taste saline, slightly bitter. Somewhat hygroscopic. Soluble at 15° in 1.2 parts of water and in 13 parts of alcohol; in half its weight of boiling water, and in 11 parts of boiling alcohol. The water-solution should be neutral or only faintly alkaline to test-paper.

SODIUM CARBONATE.

SODII CARBONAS.

$$Na_2CO_3.10H_2O=286.$$

By far the greater portion of *sal sodae*, or crude sodium carbonate, consumed at this time is prepared by the so-called *Leblanc's method.* A mixture of sodium sulphate, chalk and coal is strongly heated in a reverberatory furnace; the mass is then leached out with water, the solution concentrated, and the carbonate, collected as it separates from the hot liquid. The product contains carbonate, sulphide, hydrate, sulphate, and hyposulphite

of sodium.. The residue obtained by evaporating the first solution to dryness is called *soda-ash,* of which several hundred million pounds are annually imported into the United States. The crude sodium carbonate is purified by re-crystallization.

At Natrona, Pa., near Pittsburg, large quantities of soda are made from *cryolite,* a mineral composed of the fluorides of sodium and aluminum. A mixture of powdered cryolite and chalk is ignited in a furnace, the first reaction being: $6NaFl.Al_2Fl_6+6CaCO_3=Na_6O_6Al_2+6CaFl_2+6CO_2$. The fused mass is powdered and the sodium aluminate leached out by water. A stream of carbonic oxide is conducted through the solution of sodium aluminate, which produces sodium carbonate and aluminium hydrate: $Na_6O_6Al_2+3CO_2+3H_2O=3Na_2CO_3+Al_2(OH)_6$.

A third method consists in treating a solution of sodium chloride with NH_3 and afterwards with CO_2; the ammonium bicarbonate gives, with the NaCl, sodium bicarbonate and NH_4Cl. The sodium bicarbonate is then converted into carbonate by heat, after which the product is dissolved and crystallized.

Both the ammonia process and the cryolite process yield purer products than the sodium carbonate made by Leblanc's process.

Uses. Vast quantities of sal sodæ are used in the arts and manufactures and in the household economy. In pharmacy it is used in the preparation of various carbonates, and for the production of sodium compounds.

Purification. Commercial sodium carbonate ("sal sodæ") may be purified by recrystallization, as follows:

Soda ash	4 parts
Water	9 parts

Mix and heat to 80° until the soda ash is dissolved. Filter. Evaporate the filtrate to a density of 1.25. Let the liquid slowly cool to about 12°. Collect the crystals, drain, and dry them, and place them in a dry bottle.

Additional crops of crystals are to be recovered from the mother-liquor on evaporation.

Notes. To prevent the formation of lumps the soda ash, in

powder, should be added gradually to the water, stirring constantly.

Should the product be found to contain sulphates or chlorides, or both, it must be re-crystallized several times, as follows:

Recrystallization.

Washing soda	2 parts
Water	3 parts

Heat the water to about 40° C., and dissolve the soda in it. Filter the solution, and set it in a crystallizer that crystals may form by the spontaneous evaporation of the water at the ordinary room temperature. Collect and drain the crystals, press them gently between cloths or bibulous paper, dry hastily, put the product in dry bottles and close tightly.

Preparation of Pure Sodium Carbonate.

It may be made by heating *pure* sodium bicarbonate in a silver dish at low red heat until converted into normal carbonate, dissolving the mass in hot water, filtering, and crystallizing.

The preparation of purified sodium bicarbonate is described under that title.

Another Method.

Sal sodæ	145 parts
Oxalic acid	60 parts
Water.	

Dissolve the sal sodæ in 100 parts of hot water and the oxalic acid in another equal amount of the same solvent. Mix the solution. Let cool. Collect the sodium oxalate, wash it with 500 parts of cold distilled water on a funnel, drain, and dry. Heat to low red heat until the oxalate is decomposed. Dissolve, filter, and crystallize.

Description.—Sodium carbonate consists of large, transparent, colorless crystals; odorless; taste strongly alkaline. Efflorescent in dry air. Soluble in 1.6 parts of water at 15°, in 0.09 part at 38°, and in 0.2 part of boiling water. Insoluble in alcohol and in ether. Soluble in 1.02 parts of glycerin. Dissolves in its own water of crystallization at 32°.5, and begins to lose some of that water. The water-solution has a strongly alkaline reaction.

Dried Sodium Carbonate.

$Na_2CO_3.2H_2O$.

Expose crystallized sodium carbonate in small fragments or crystals, for several days, to a temperature not exceeding 25° until completely effloresced; then dry it at not over 45° until reduced to one-half the weight of the crystallized salt originally taken.

Triturate the product in a mortar until a uniform powder is obtained and keep this in well-closed bottles.

Notes. Crystallized sodium carbonate contains 10 molecules of water of crystallization. When exposed to the air it effloresces, and in the course of one or more weeks, according to the temperature and dryness of the air, it loses about 40 per cent of its weight. At 45° C. it loses all but 2 molecules of its water of crystallization. [At 90° to 100° C. it becomes anhydrous.]

At about 32.°5 crystallized sodium carbonate dissolves in its water of crystallization. This "aqueous fusion" should be avoided in the preparation of dried sodium carbonate because it renders the subsequent completion of the process more difficult. It is, therefore, directed that the crystallized salt shall be effloresced at from 20° to 25° C. before higher heat is applied.

Description.—A white, amorphous powder.

SODIUM CHLORATE.

SODII CHLORAS.

$NaClO_3$=106.25.

Prepared from calcium hydroxide and sodium chloride with chlorine, or from chlorinated lime and sodium chloride, by a process analogous to that for preparing potassium chlorate.

It may also be made as follows:

Tartaric acid	117 parts
Sodium carbonate	110 parts
Potassium chlorate	96 parts

Dissolve the acid and the carbonate in 1000 parts of hot water.

To the hot solution add the potassium chlorate. Potassium bitartrate separates, and the solution contains the sodium chlorate together with some potassium bitartrate, which deposits during the evaporation to crystallization, the last being gotten rid of by crystallizing it out from the saturated solution of the sodium chlorate.

Description.—Colorless transparent crystals, or a white crystalline powder; odorless; taste saline, cooling. Soluble at 15° in 1.1 parts of water, and in about 100 parts of alcohol; in half its weight of boiling water and in 40 parts of boiling alcohol; in 5 parts of glycerin. The water-solution is neutral to test-paper. Like potassium chlorate the sodium chlorate must be handled with care, and not triturated with reducing agents or subjected to concussion.

SODIUM CHLORIDE.

SODII CHLORIDUM.

$NaCl=58.4$.

Diluted hydrochloric acid	5 parts
Sodium carbonate	2 parts

Add the sodium carbonate gradually to the diluted acid. Make the solution quite neutral to litmus paper. Filter. Evaporate to dryness, or to crystallization, as may be desired.

Reaction. $Na_2CO_3+2HCl=2NaCl+H_2O+CO_2$.

Purification of Common Salt.

Common salt	1 kilogram
Water	3½ liters
Lime	12 Gm
Barium chloride,	
Sodium carbonate,	
Hydrochloric acid.	

Dissolve the salt in the water. Slake the lime, mix it with 100 ml of water, and add the mixture to the salt solution. Heat to the boiling point. Filter. Add a solution of barium chloride to the filtrate as long as it causes precipitation. Then add solution of sodium carbonate until all the calcium and barium shall have been

separated. Filter. Neutralize with hydrochloric acid. Evaporate to crystallization or granulation, and dry the salt thoroughly.

Notes. Common salt contains magnesium and calcium salts (chlorides and sulphates). These are precipitated as described.

Description.—Colorless crystals or a white, granular crystalline powder; dry; odorless, and of purely saline taste. Not hygroscopic. Soluble in 2.8 parts of water at 15°, and in 2.5 parts at 100°. Almost insoluble in alcohol. The water-solution is neutral to litmus paper.

Kissingen Salt.

Dried sodium sulphate	90 parts
Precipitated calcium sulphate	50 parts
Potassium sulphate	11 parts
Ferrous sulphate	3 parts
Sodium bicarbonate	170 parts
Dried magnesium sulphate	130 parts
Sodium chloride	400 parts
Tartaric acid	10 parts

Mix the separately powdered dry ingredients well.

SODIUM CITRATE SOLUTION.

LIQUOR SODII CITRATIS.

A solution of normal sodium citrate of which each milliliter contains 0.50 Gm of $Na_3C_6H_5O_7$ is prepared as follows:

Citric acid	400 Gm
Sodium bicarbonate	486 Gm

Dissolve the sodium bicarbonate in 750 ml of distilled water by the aid of heat, gradually add the citric acid, and, when effervescence has ceased, bring the liquid to the boiling point. If the solution is not neutral to litmus paper, add more sodium bicarbonate (or citric acid, as the case may require) to produce a perfectly neutral reaction. Finally, add enough distilled water to make the whole measure 1080 ml.

To make solution of *di-sodium-hydrogen citrate,* containing

0.50 Gm of $Na_2HC_6H_5O_7$ in each ml add 200 Gm of citric acid to 1080 ml of the above solution, and finally add enough distilled water to make the whole product measure 1480 ml.

Used in making certain scale salts of iron.

Potio Riveri

is a preparation of the German Pharmacopœia made of 4 parts of citric acid, 190 parts of water and 9 parts of sodium carbonate in crystals. It is directed to be freshly prepared whenever prescribed.

SODIUM ETHYLSULPHATE.

SODII ET AETHYL SULPHAS.

[Sodium Sulphovinate.]

$NaC_2H_5SO_4.H_2O=166.$

Alcohol	5 parts
Sulphuric acid	5 parts
Barium carbonate	10 parts
Sodium carbonate	3 parts

Put the alcohol in a large porcelain dish, and set it in rapid rotary motion by stirring with a glass rod. Add the sulphuric acid in a small stream. Pour the cooled mixture into a bottle and close it with a glass stopper. Let it stand over night. Pour it back into the porcelain dish. Add gradually, and with constant and vigorous stirring, the powdered barium carbonate. previously sifted so as to be free from lumps. After all the barium carbonate has been added return the mixture to the bottle and shake well. Filter the liquid. Then add to it, in small portions at a time, the sodium carbonate until no further precipitation occurs on the addition of more. [Filter a small portion of the liquid before all of the sodium carbonate has been added, and test it by further addition of sodium carbonate to determine how much more of the alkali carbonate is required to precipitate the barium.] When all the barium has been precipitated [without using an excess of sodium carbonate], filter the solution and evaporate the filtrate with the aid of moderate heat to crystallization.

Description.—Colorless laminar crystals, somewhat efflorescent; readily soluble in water.

Solution of Sodium Ethylate; B. P.

An alcoholic solution containing 18 per cent of C_2H_5ONa.

Sodium, clean and bright	1 Gm
Absolute alcohol	20 ml

Cautiously dissolve the sodium in the alcohol in a flask kept cool by flowing water.

Reaction. $2C_2H_5OH+Na_2=2C_2H_5ONa+H_2$.

Notes. The metal should, if larger quantities be employed, be added gradually. The heat liberated by the reaction is great, and hence the liquid must be kept cold.

Description.—A colorless syrupy liquid which becomes brown on keeping. It must be freshly prepared when required. Sp. w. 0.867.

SODIUM HYDROXIDE.

SODII HYDROXIDUM.

NaOH=40.

Prepared by evaporating the solution of sodium hydroxide to dryness, fusing the residue, dissolving it in alcohol, which leaves carbonate undissolved, after which the alcohol is distilled off from the clear solution and the sodium hydroxide again fused.

This alkali must be preserved in the same manner as potassium hydroxide—in tightly closed bottles of hard green glass, the stoppers to be rubbed with a little petrolatum to prevent them from being cemented into the necks of the bottles.

Description.—Dry, white, translucent, hard, of crystalline fracture, odorless, acrid, caustic, corrosive. Hygroscopic, but not deliquescent, becoming first moist and then converted into dry carbonate on exposure to air. Soluble in 1.7 parts of water at 15°, and in 0.8 part of boiling water. Freely soluble in alcohol. Strongly alkaline.

SODIUM HYDROXIDE SOLUTION.

LIQUOR SODII HYDROXIDI.

[Liquor Sodæ; U. S.]

An aqueous solution containing 5 per cent of sodium hydroxide, $NaOH=40$.

Sodium carbonate	17 parts
Calcium oxide	5 parts
Distilled water, sufficient.	

Dissolve the sodium carbonate in 40 parts of boiling distilled water. Slake the lime and make of it a smooth mixture with 40 parts of distilled water and heat this mixture to boiling. Then add gradually the sodium carbonate solution to the "milk of lime," and boil the whole mixture for ten minutes. Remove the heat, cover the vessel tightly, and, when the contents are cold, add enough distilled water to make the whole weigh 100 parts. Lastly strain the liquid through linen, or, when the precipitate has subsided, draw off the clear solution by means of a syphon.

Reaction. $Na_2CO_3+Ca(OH)_2=2NaOH+CaCO_3$.

Notes. The sodium carbonate employed must, of course, be the crystallized salt, not in any degree effloresced.

The lime might advantageously be washed free from dust and salts before being used. This is done as follows: Slake the 5 parts of lime with about 40 parts of water; then add about 500 parts of water and stir the mixture occasionally during half an hour; let the calcium hydroxide subside, decant the liquid and throw this away. Then add 40 parts of pure water, heat to boiling and use this liquid with the solution of sodium carbonate as described in the formula above given.

A considerable excess of lime is used, but as calcium hydroxide is insoluble in a solution of sodium hydroxide no lime will be contained in the product. An unnecessarily large excess of lime is, however, objectionable because the superfluous calcium hydroxide does not subside as readily as the calcium carbonate.

If cold liquids are used the calcium carbonate formed by the reaction is so finely divided and light that it subsides very slowly

and the bulky precipitate retains much liquid causing corresponding loss of product.

More concentrated solutions can not be used because the reaction will not then be complete. It is completed when the liquid no longer effervesces with dilute acid or when it does not cause turbidity when dropped into a clear solution of calcium hydroxide.

Solution of sodium hydroxide is usually clarified by subsidence and decantation; but it may, if preferred, be successfully filtered through coarsely powdered and washed marble.

The strength of such a preparation as solution of sodium hydroxide should not be fixed by the quantities and proportions of the materials as is done in the Pharmacopœia from which the foregoing formula is borrowed; it should be tested quantitatively with volumetric solution of sulphuric acid and the strength adjusted by dilution in accordance with the result. In order to carry out this plan the quantity of water used in the process may be diminished ten per cent and the product diluted as required; or the amount of water directed may be used and the product diluted, or concentrated by evaporation, as may be found necessary.

Solution of sodium hydroxide attacks organic matter energetically and is therefore discolored by contact with organic substances. It also attacks ordinary flint glass so that if it be put in glass-stoppered bottles of that kind of glass the stoppers usually become firmly fixed and can not be removed, necessitating the breaking of the bottles to get at the contents. The solution should, therefore, be kept in bottles made of green glass which is not so easily attacked by the alkali, and the stoppers should be coated with a little paraffin or petrolatum.

Description.—The official sodium hydroxide solution is a clear, colorless and odorless liquid, having a very acrid and caustic taste, a corrosive action on organic matter, and a strongly alkaline reaction.

Its specific weight is about 1.059, and the specific volume 0.944.

Valuation. To neutralize 20 Gm of official solution of sodium hydroxide should require 25 ml of normal sulphuric acid, each ml of the test solution indicating 0.2 per cent of NaOH; phenolphtalein should be used as indicator.

One ml of normal sulphuric acid is the equivalent of 0.03996 Gm of NaOH.

SODIUM HYPOCHLORITE SOLUTION.

LIQUOR SODAE CHLORATAE.

[Labarraque's Solution.]

An aqueous solution containing sodium hypochlorite as its most important constituent. The Pharmacopœia requires that the preparation shall furnish at least 2.6 per cent of available chlorine.

Sodium carbonate	30 parts
Chlorinated lime	15 parts
Water, sufficient.	

Triturate the chlorinated lime with 40 parts of water, producing a uniform mixture. Let the heavier particles subside and transfer the liquid to a filter. Treat the undissolved solid matter with 35 parts of water and add this mixture to the first, passing the whole through the filter. Wash the filter and contents with 20 parts of water, letting this portion also pass through the filter and be added to the previous solutions.

Dissolve the sodium carbonate in 80 parts of boiling water. Add this solution to the united filtrates obtained from the chlorinated lime mixtures. Stir the mixture thoroughly, and, if it should become gelatinous, apply heat until liquefaction is effected. Transfer the mixture to a new filter, collect the filtrate and add as much more water, through the filter, as may be necessary to make the total product 200 parts.

Keep the solution in well-stoppered bottles, protected from light and in a cool place.

Reaction.

$$Ca(OCl)_2+CaCl_2+2Na_2CO_3=2NaOCl+2NaCl+2CaCO_3.$$

Notes. The object of using hot solution of sodium carbonate is to produce a dense calcium carbonate, which settles more readily than a lighter precipitate. At the same time it should be remembered that high heat decomposes the hypochlorite of so-

dium formed, and the value of the preparation would then be impaired by loss of available chlorine. On the addition of acids the solution effervesces because carbon dioxide and chlorine are liberated; the CO_2 comes from the excess of sodium carbonate. The containers should be closed with rubber stoppers or glass stoppers instead of corks, as the chlorine destroys the latter.

Description.—A pale-greenish clear liquid, having a faint odor of chlorine, and an alkaline as well as salty and peculiar taste. Sp. w. about 1.052. Colors red litmus paper blue and then bleaches it. On the addition of hydrochloric acid the liquid gives off chlorine and carbon dioxide.

SODIUM HYPOPHOSPHITE.

SODII HYPOPHOSPHIS.

$NaPO_2H_2.H_2O=106.$

Calcium hypophosphite.....	6 parts
Sodium carbonate.....	10 parts

Dissolve each salt in 60 parts of boiling water, and mix the solutions. Filter out the calcium carbonate, and evaporate the solution over a water-bath until a dry granular salt remains, taking care that the temperature during the evaporation does not exceed 85° C.

Notes. May be purified by re-dissolving it in ten times its weight of hot alcohol, filtering, and evaporating to dryness.

Reaction. $Ca(H_2PO_2)_2+Na_2CO_3=CaCO_3+2NaH_2PO_2.$

Description.—Pearly white or colorless crystals, or a white granular powder; odorless; taste saline, bitterish. Hygroscopic. Soluble at 15° in its own weight of water and in 30 parts of alcohol; in 0.12 part of boiling water and in its own weight of boiling alcohol. Insoluble in ether. The water-solution is neutral to litmus paper.

SODIUM IODIDE.

SODII IODIDUM.

NaI=149.5.

Iron wire, cut	6 parts
Iodine	20 parts
Sodium carbonate	24 parts
Distilled water, sufficient.	

Digest the iron and 15 parts of the iodine with 50 parts of water in a flask until the odor of iodine ceases and a green solution of ferrous iodide results. Filter this, and dissolve the remainder of the iodine in the filtrate. Dissolve the sodium carbonate in 80 parts of water, heat to boiling, then add the solution of iodides of iron during constant stirring, boil the mixture fifteen minutes, filter, evaporate, and crystallize.

Notes. The reactions are analogous to those taking place in the process for preparing potassium iodide in a similar way. See the notes under that title.

Sodium iodide may also be made from iodine and solution of sodium hydroxide, as potassium iodide is prepared from iodine and solution of potassium hydroxide.

Description.—Colorless crystals or a white crystalline powder; odorless; taste saline, somewhat bitterish. Hygroscopic. Decomposes when exposed to the air, forming sodium carbonate and free iodine which discolors the product. Soluble at 15° in 0.6 part of water and in about 3 parts of alcohol; in one-third its weight of boiling water and in 1.4 parts of boiling alcohol. The water-solution should be neutral or only faintly alkaline to test-paper.

SODIUM NITRATE.

SODII NITRAS.

$NaNO_3$=85.

Crude sodium nitrate occurs native in Peru and Bolivia. The native sodium saltpeter contains from 20 to 80 per cent of so-

dium nitrate, most frequently yielding 50 per cent. It is freed from earth and other impurities by solution, straining, and crystallization.

It is purified in the same way as potassium nitrate, by recrystallization and by washing with a cold saturated solution of pure sodium nitrate, which removes chlorides present.

It can be prepared by the neutralization of diluted nitric acid with sodium carbonate.

Description.—Colorless transparent crystals; odorless; taste saline, cooling, bitterish. Hygroscopic. Soluble in 1.3 parts of water and in 100 parts of alcohol at 15°; in 0.6 part of boiling water and in 40 parts of boiling alcohol. The water-solution is neutral to litmus paper.

SODIUM NITRITE.

SODII NITRIS.

$NaNO_2$=69.

Prepared by fusing together sodium nitrate and sheet lead, keeping the two substances together at the temperature required for fusion until the lead oxidizes to lead oxide at the expense of the sodium nitrate which is thus reduced to sodium nitrite. The nitrite is leached out from the cooled mass with water and the solution treated with carbon dioxide to free it from lead, after which the liquid is filtered and evaporated to crystallization.

Description.—Colorless crystals fused into white masses or sticks; odorless; taste mild, saline. Hygroscopic. Oxidizes in air to nitrate. Soluble at 15° in 1.5 parts of water. Freely soluble in boiling water. Sparingly soluble in alcohol. The water-solution has an alkaline reaction on test-paper.

SODIUM NITROPRUSSIDE.

SODII NITROPRUSSIDUM.

$Na_2Fe(CN)_5NO.2H_2O$=300.

Potassium ferrocyanide	1 part
Nitric acid (69% HNO_3)	2 parts
Sodium carbonate, anhydrous, about	2 parts
Water.	

Reduce the ferrocyanide to fine powder. Dilute the nitric acid

with about 1½ parts of water in a flask. Add the powder in small portions to the acid, shaking after each addition, and waiting until effervescence ceases before adding more. When all of the ferrocyanide has been added heat the mixture at full water-bath heat for one hour, or until a test sample of the liquid no longer gives a precipitate on addition of a solution of ferric chloride. Then set the flask aside in a cool place for about twelve hours. Decant the green liquid from the crystals of potassium nitrate. Neutralize the liquid with *a sufficient quantity* of sodium carbonate added in small portions. Heat the liquid to the boiling point to expel any carbonic acid, and then add a concentrated *solution* of sodium carbonate, in small portions, stirring well, until the liquid becomes quite neutral again, but avoiding an excess of the alkali carbonate. Filter while hot. Evaporate the filtrate in a porcelain dish until a large quantity of crystals has separated. Pour off the hot mother-liquor. Collect and drain the crystals, and then dry them over sulphuric acid.

Evaporate the mother-liquor to obtain an additional crop of crystals out of the hot liquid and collect, drain, and dry them as before, avoiding the deposit of any nitrate.

Redissolve the crystals in twice their weight of hot water, filter into an evaporation dish and set this over sulphuric acid in a desiccator, in a shady place. Change the sulphuric acid as often as may be necessary.

Notes. Slow evaporation is necessary to obtain large crystals. The sulphuric acid should be changed every two or three days. At all stages of the process the product must be protected against strong light.

Properties. Large, fine, blood-red crystals, soluble in 2.5 parts of water. Decomposed by light when in solution.

SODIUM OLEATE.

SAPO MEDICINALIS DURUS.

OLIVE OIL SOAP.

["Medicinal Soap."]

Olive oil	100 parts
Sodium hydroxide	15 parts
Distilled water	35 parts
Alcohol	30 parts

Dissolve the sodium hydroxide in the water, add the oil and the alcohol, and heat the mixture gently, in a porcelain dish, over a water-bath, stirring constantly, until saponification is completed. Then add 300 parts of warm distilled water and mix well.

Make a solution of—

Sodium chloride	25 parts
Sodium carbonate	5 parts
Distilled water	80 parts

and filter it. Add this solution to the solution of the soap, and mix thoroughly.

Heat the mixture over the water-bath, stirring gently, until the soap separates and rises to the surface of the liquid. Let the dish and contents cool. When cold separate the mother liquor from the soap, and rinse the soap with a little cold water. Then squeeze the water out of the soap by means of strong pressure, dry the mass with the aid of gentle heat, and powder the product.

Notes. The quantity of sodium hydroxide prescribed is slightly in excess of the amount theoretically required. All hard soaps consist mainly of sodium oleate. The best Castile soap approaches pure sodium oleate sufficiently for all practical purposes in pharmacy, including the preparation of other oleates by "double decomposition." No soda soap should be used for the preparation of other metallic oleates except after complete drying; hence *powdered* soap is to be used. This is because the amount of moisture in powdered soap can not vary a great deal.

The soap is completely soluble in water although the solution is not clear. Soda soap is precipitated from its water solution by sodium chloride in the manner described.

Transparent soap may be made by dissolving soda soap in alcohol, filtering the solution, and evaporating. Castor oil soap easily forms a transparent soap.

Medicinal soda soaps are sometimes made from almond oil, and also from fresh (unsalted) butter.

SODIUM PHENOLSULPHONATE.

SODII PARAPHENOLSULPHONAS.

(Sodium Sulphocarbolate.)

$NaC_6H_5SO_4.2H_2O=232.$

Crystallized phenol	10 parts
Sulphuric acid	12 parts
Barium carbonate	20 parts
Sodium carbonate	34 parts

Add the phenol to the sulphuric acid, stir, and heat the mixture at 55° C. for several days. Add 200 parts of water, and stir well. Then gradually add the barium carbonate, and mix the whole intimately. Filter. Precipitate the filtrate exactly with the sodium carbonate, previously dissolved in 100 parts of water. Again filter, and evaporate to crystallization. Purify the product by re-crystallization until colorless, or but faintly pinkish.

Reaction.

$2HC_6H_5SO_4+BaCO_3=Ba(C_6H_5SO_4)_2+H_2O+CO_2$; and then

$Ba(C_6H_5SO_4)_2+Na_2CO_3=2NaC_6H_5SO_4+BaCO_3$.

Notes. Any excess of sulphuric acid present in the phenol-sulphonic acid is removed by the use of an excess of barium carbonate, whereby insoluble barium sulphate is formed, which is filtered away.

Description.—Colorless, transparent, rhombic prisms, permanent in the air, odorless or nearly so, having a cooling, saline, somewhat bitter taste, and a neutral reaction. Soluble in 5 parts of water, and in 132 parts of alcohol at 15° C. (59° F.); in 0.7 part of boiling water and in 10 parts of boiling alcohol.

SODIUM PHOSPHATE.

SODII PHOSPHAS.

$Na_2HPO_4.12H_2O=358.$

[Di-Sodium-Hydrogen Phosphate.]

Bone-ash, in fine powder	10 parts
Sulphuric acid	6 parts
Sodium carbonate, sufficient.	

Wash the bone-ash with ten times its weight of boiling water. Decant and reject the water. Mix the wet mass with the acid, set the mixture in a warm place for three days, replacing the water vaporized by the heat, and stir occasionally. Then add 10 parts of boiling water, mix thoroughly, transfer the mixture to a muslin strainer, and let the liquid pass through. Reserve the liquid. Transfer the magma from the muslin to an earthenware vessel, add 10 parts of boiling water, stir well, and again strain the mixture through muslin, adding the colature to that previously reserved. Continue washing the residue with boiling water until the liquid passes nearly tasteless. Mix the several acid liquids and evaporate the mixture to 20 parts. Set this aside a few days. Filter. Heat the filtrate to the boiling point, and gradually add to it a solution of sodium carbonate in hot water, until a filtered portion of the now alkaline liquid effervesces when mixed with dilute sulphuric acid. Digest the mixture an hour or two, and then filter. Wash out the remaining sodium phosphate from the residue by means of hot water, adding the washings to the filtrate. Then evaporate the mixed liquids to 10 parts, and set aside to crystallize.

Purify the product by re-crystallization after dissolving it in twice its weight of water. Repeat the re-crystallization as many times as may be necessary to obtain a clean product.

Dry the crystals with as little exposure to the air as possible, to avoid efflorescence.

Reaction. First the tri-calcium phosphate (bone-ash) is decomposed by the sulphuric acid yielding acid calcium phosphate and calcium sulphate as follows:

$$Ca_3(PO_4)_2+2H_2SO_4=CaH_4(PO_4)_2+2CaSO_4.$$

Then the acid phosphate of calcium is decomposed by the sodium carbonate as follows:

$$CaH_4(PO_4)_2+2Na_2CO_3=2Na_2HPO_4+CaCO_3+CO_2+H_2O.$$

Notes. When the bone-ash is treated with sulphuric acid the mixture becomes hot. Usually the powdered bone-ash is not washed previous to mixing it with the sulphuric acid, and no water added until after the powder and acid have first been thoroughly mixed. If organic matter be present in the bone-ash, sul-

phur dioxide is given off. Some carbon dioxide will also be given off from calcium carbonate in the bone-ash. If the bone-ash is washed with water, the object of which is the removal of alkalies, the wash-water should be drained off thoroughly before mixing the moist mass with the acid. When the process is carried out on a small scale the washed and drained bone-ash, still wet, should be put in a tared vessel, the sulphuric acid mixed with it, and finally enough boiling water added to make the whole mixture weigh three times as much as the bone-ash used. After the mass has been digested a sufficient time to insure thorough decomposition of the basic calcium phosphate, to facilitate which the mass should be frequently and thoroughly stirred, the next step is the separation of the liquid containing the acid phosphate of calcium, with which the sodium phosphate is to be made, from the white mass of calcium sulphate which is to be thrown away after the acid liquid has been completely washed out from it and collected.

On a manufacturing scale this leaching process may be conveniently carried out in tubs provided with loose perforated diaphragms resting on suitable supports about four or five inches above their bottoms. A common tight barrel sawed in two in the middle furnishes two good tubs. A hole is bored in one side of each tub, about two inches above the bottom, and a tube about six inches in length is fitted tightly in the hole. A piece of very coarse, loose gunny-bagging is washed in warm water and, while still wet, spread out over the perforated false bottom so as to cover it and extend two or three inches up on the sides of the tub all around. The mixture of bone-ash, acid and water is now put into the tub, which should be about half filled. A stick of wood four feet long furnishes a good stirrer. Boiling water is now poured upon the mass and well mixed with it, after which the liquid is allowed to percolate through the mass, and through the strainer, into the space between the perforated diaphragm and the bottom of the tub, and to run out through the tube into a bucket, or other suitable vessel.

When the liquor which passes from the tub is nearly tasteless, or no longer acid, the leaching is completed, the residue of calcium sulphate is thrown away, and the acid liquor is boiled down to twice the weight of the bone-ash used.

The concentrated solution of acid calcium phosphate is now

allowed to stand a few days in order that the calcium sulphate held in solution may be deposited. It is then filtered, diluted with an equal volume of water, and heated to the boiling point. To the hot liquid is now added hot sodium carbonate solution as described.

The quantity of sodium carbonate required will depend upon the completeness of the decomposition of the bone-ash and the lixiviation—in other words, upon the amount of acid calcium phosphate contained in the liquor. If the work has been well done, it will require about 100 parts or more of sodium carbonate to render alkaline the acid liquor from 100 parts of bone-ash.

Hot liquids are employed in order that the calcium carbonate formed may be heavy, so as to separate as rapidly as possible. The sodium carbonate must be added gradually, because of the effervescence caused by the escaping carbonic oxide. Litmus paper cannot be used to ascertain when sufficient sodium carbonate has been added, because sodium phosphate itself has an alkaline reaction. After the further addition of sodium carbonate has ceased to cause effervescence, it is necessary to remove a small portion of the liquid, filter it, and test it by pouring it into a small amount of dilute sulphuric acid, when it should effervesce. The slight excess of sodium carbonate added is necessary to insure satisfactory crystallization of the sodium phosphate, and the carbonate remains in the mother-liquor.

When the liquid contains an excess of sodium carbonate, the precipitated calcium carbonate is filtered out and washed on the filter with a little hot water, the washings being added to the previous filtrate. Evaporate the filtrate until reduced to about three times the weight of the sodium carbonate consumed, or until a pellicle begins to form on the surface. Then set it aside to crystallize.

The crystallization is best effected when an excess of sodium carbonate is present and the solution is not too concentrated. It should not be hastened by evaporating down the solution too far, because the salt then crystallizes with only 7 instead of 12 molecules of water of crystallization.

The product generally requires purification by recrystallization to obtain a sodium phosphate which answers the description and tests of the Pharmacopœia.

Description.—Colorless transparent crystals, having a specific

gravity of 1.55. They effloresce readily, and when slightly heated melt in their water of crystallization.

The salt is odorless, and has a cooling, saline, somewhat alkaline, taste. Efflorescent.

Soluble in 5.8 parts of water at 15°, and in 1.5 parts of boiling water. Insoluble in alcohol. The water-solution is alkaline to litmus paper, but not to phenolphtalein paper.

Recrystallized Sodium Phosphate.

Sodium carbonate	1 part
Commercial sodium phosphate	50 parts
Water	250 parts

Dissolve the sodium phosphate in the water with the aid of heat, filter the solution, put it in a crystallizer and set this aside at perfect rest in a place free from dust that crystals may form by spontaneous evaporation of the solvent at the ordinary room temperature.

Should crystallization be much delayed place a few clear crystals of pure sodium phosphate in the solution to start it.

Collect the crystals in the usual way, drain them, press them gently between cloths or bibulous paper, dry them hastily, and at once put the product in dry bottles, which must be tightly closed and kept in a cool place.

Dried Sodium Phosphate.

$Na_2HPO_4 = 142.$

Heat crystallized sodium phosphate in porcelain dish over a water-bath until it ceases to lose weight, stirring the contents until dry. Then heat the powder on a sand-bath at about 120° C. for a few minutes. Keep the product in a tightly-closed bottle.

Notes. Crystallized sodium phosphate contains 12 molecules of water of crystallization. When heated to 35° C. it begins to liquefy, but does not completely dissolve until at about 40° C. If then allowed to cool at once it solidifies to a cake of a radiated crystalline texture.

When heated above 40° C. the salt loses 5 molecules of water.

At 100° C. all the water of crystallization is finally expelled and "dried sodium phosphate" remains. But as water-bath heat

is not sufficient (being less than 100° C.) to effect this result, the drying must be finished with the aid of the sand-bath.

"Effloresced sodium phosphate," formed by the gradual drying of the crystallized salt in dry warm air, contains 47 per cent of water, or seven molecules, and the "dried sodium phosphate" (anhydrous) gradually absorbs moisture, when exposed to the air, until converted into $Na_2HPO_4.7H_2O$.

Description.—A white, amorphous powder.

Sodium Ammonium Phosphate.

SODII ET AMMONII PHOSPHAS.

[Microcosmic Salt.]

$NH_4NaHPO_4.4H_2O=418.$

Sodium phosphate	5 parts
Ammonium phosphate	2 parts

Dissolve the salts in 20 parts of hot water, add a little ammonia so as to render the liquid alkaline to turmeric paper, filter, and set aside to crystallize.

The crystals effloresce; their solution has a slightly alkaline reaction.

The salt is used as a reagent in blow-pipe analysis.

SODIUM PYROPHOSPHATE.

SODII PYROPHOSPHAS.

$Na_4P_2O_7.10H_2O=446.$

Prepared by heating sodium phosphate in an iron dish, raising the heat gradually until the salt fuses in its water of crystallization, then falls to powder as that water is expelled, and finally decomposes at a temperature of 240° to 300° C., being converted into normal sodium pyrophosphate. The dry powder is dissolved in a sufficient quantity of boiling water—about five times its weight—the solution is filtered while hot, and then set aside to crystallize. More crystals are to be obtained from the mother liquor upon evaporation.

Reaction. $2Na_2HPO_4=Na_4P_2O_7+H_2O.$

Notes. Sodium phosphate, in solution, yields a yellow precipitate with test-solution of silver nitrate acidulated with nitric acid; sodium pyrophosphate yields a perfectly white precipitate with the same reagent. The conversion of the phosphate is, therefore, known to be completed when a sample of the dry powder, dissolved in water, yields a white precipitate with silver nitrate.

From 716 parts of sodium phosphate, the yield of pyrophosphate of sodium should be 446 parts, or nearly 62 per cent.

Description.—Colorless, transparent crystals, odorless, of cooling, saline, somewhat alkaline, taste. Soluble in 12 parts of water at 15° and in 1.1 parts of boiling water. Insoluble in alcohol. The water-solution has a slightly alkaline reaction on both litmus paper and phenolphtalein.

SODIUM SALICYLATE.

SODII SALICYLAS.

$NaC_7H_5O_3$=160.

Salicylic acid	10 parts
Sodium bicarbonate	6 parts
Water	40 parts

Shake the salicylic acid well with the water so as to distribute it uniformly through the liquid. Then add the sodium bicarbonate, a little at a time, shaking well after each addition. Warm the liquid slightly to expel the carbonic acid which is set free. When cold again, filter the solution, and evaporate at not over 60° C. to dryness, stirring constantly so as to obtain a granulated product. Keep it in well stoppered bottles in a cool place and protected against light.

Reaction. $2HC_7H_5O_3+2NaHCO_3$

$=2NaC_7H_5O_3+2H_2O+2CO_2$.

Notes. It is important that salicylic acid should be in excess throughout; consequently a small excess of the acid is used, and the sodium bicarbonate added to the acid instead of vice versa. Should the alkali be in excess, the product may become darkened even during the process of manufacture, and never fails to be

dark-colored when finished or after being kept a short time.

E. Hoffman recommends preparing the salt by stirring 20 parts of sodium bicarbonate and 33 parts of dialyzed salicylic acid with enough water to form a thick paste, allowing the carbonic acid time to escape, and then evaporating to dryness.

The presence of iron (even mere traces) causes a reddish discoloration of the salt and solutions of it. Hence filter paper quite free from iron must be used.

A crystallized product may be obtained from a saturated alcoholic solution. The crystals should then be drained in a funnel and dried in a porcelain dish with the aid of moderate heat. An additional quantity of salt may be recovered from the mother-liquor on evaporation. Should the mother-liquor be colored it may be decolorized by filtration through animal charcoal unless the coloration is due to iron. The salicylic acid contained in the last mother-liquor can be recovered by evaporating to dryness, redissolving the residue in water and precipitating by adding an excess of diluted sulphuric acid.

Description.—A white, amorphous or crystalline powder, odorless, sweetish, saline, afterwards acrid. Soluble in 0.9 part of water, and in 6 parts of alcohol at 15°. Very soluble in these solvents at their boiling points. Soluble also in glycerin. The water-solution should have a slightly acid reaction on test-paper.

SODIUM SANTONINATE.

SODII SANTONINAS.

$$2NaC_{15}H_{19}O_4.7H_2O = 698.$$

Santonin	10 Gm
Solution of soda	35 Gm
Water	10 Gm

Mix and digest until dissolved; filter, set in a cool place to crystallize. Evaporate the mother-liquor to recover more, as long as the crystals obtained are colorless.

Keep it in a tightly-corked bottle, and protected from the light.

Recover the remainder of the santonin from the last mother-

liquor by acidulating with hydrochloric acid, when it will separate in crystalline form.

Albuminated Santoninate of Sodium.

Santonin	10 Gm
Sodium bicarbonate	40 Gm
Dry soluble albumen	20 Gm
Water	500 ml

Digest together at 60° C. until dissolved. Filter. Evaporate by gentle heat. Spread it on glass plates to dry.

Brilliant pearly scales, soluble in water.

SODIUM SILICATE SOLUTION.

LIQUOR SODII SILICATIS.

(Water-glass. Soluble glass. Liquid glass.)

Containing 33 per cent of a mixture of sodium tri-silicate ($Na_2Si_3O_7$) and sodium tetrasilicate ($Na_2Si_4O_9$). It must be free from caustic alkali.

When an intimate mixture of one molecule of dried sodium carbonate and four molecules of silica are fused together, the fused mass is water-soluble, and a solution having a sp. w. of from 1.30 to 1.40 is employed for surgical dressings. It is a syrupy, colorless, odorless liquid.

SODIUM SULPHATE.

SODII SULPHAS.

$Na_2SO_4.10H_2O=322.$

[Glauber's Salt.]

Obtained as a bye-product in the manufacture of hydrochloric acid from sodium chloride and sulphuric acid. Also in many other chemical processes.

The crude Glauber's Salt may be purified as follows:

Crystallized Sodium Sulphate.

Glauber's salt	2 parts
Water	3 parts
Lime,	
Chlorinated lime.	

Dissolve the salt in the water and the solution to the boiling point.

Slake a piece of lime, and add water enough to form a thick, milky mixture; of this "milk of lime" add so much to the hot solution of Glauber's salt to render it alkaline to test paper. Stir well.

Mix some chlorinated lime with enough water to form a thick, milky mixture; of this add a small quantity at a time to the solution of Glauber's salt until a filtered test sample no longer becomes discolored by hydrogen sulphide after acidulation with hydrochloric acid.

When free from iron, filter the liquid, and evaporate the filtrate until it has acquired the sp. w. 1.25 at 40° C. Set the solution aside in a crystallizer, at rest, and let crystallization be effected in the usual way. Drain the crystals, press them gently between cloths or bibulous paper until dry, and at once place them in dry bottles, which are to be tightly closed.

Pure Sodium Sulphate may also be made from diluted sulphuric acid and sodium carbonate.

Description.—Large, colorless, transparent crystals, odorless; of somewhat bitter, saline taste. Efflorescent in air. Soluble at 15° in 2.8 parts of water; at 34° in one-fourth its weight of water; at 100° in 0.47 part of water. Insoluble in alcohol. Neutral.

Carlsbad Salt.

Dried sodium sulphate	22 parts
Sodium bicarbonate	18 parts
Sodium chloride	9 parts
Potassium sulphate	1 part

Mix the previously powdered dry salts.

[Carlsbad water contains about 6 Gm of salts to each liter.]

Notes. The chloride and the bicarbonate must be well dried, separately, and then powdered. The sulphate, in crystals, should also be powdered separately, which can hardly be effected otherwise than by granulating the previously dissolved salt. If dried sodium sulphate is used, the product will yield hard lumps when water is added, and these hard particles dissolve but slowly.

Effervescent Carlsbad Salt.

Artificial Carlsbad salt	100 parts
Sodium bicarbonate	100 parts
Tartaric acid	54 parts
Citric acid	36 parts

Mix the powdered ingredients in a mortar, transfer the mixture to a porcelain dish and heat it over a water-bath, stirring constantly, until a pasty mass is formed. Granulate this by passing it through a very coarse sieve, in the usual way, and dry the product at about 25° C.

Dried Sodium Sulphate.

Na_2SO_4=142.

Place the crystallized sodium sulphate in a porcelain dish over the water-bath and expose it to a moderate heat until reduced to scarcely more than 44 per cent of its original weight and completely effloresced.

Notes. Crystallized sodium sulphate contains about 56 per cent of water of crystallization. It readily parts with all of that water even at ordinary room temperatures, and effloresces very rapidly when moderately heated.

Description.—A white, amorphous powder.

SODIUM SULPHITE.

SODII SULPHIS.

$Na_2SO_3.7H_2O$=252.

Prepared by saturating a solution of sodium carbonate with sulphur dioxide, and then adding another equal quantity of sodium carbonate, after which the solution is evaporated to crystallization. The process is analogous to that for making potassium sulphite.

In both cases a bisulphite is first made, and this is converted into normal sulphite by the addition of a quantity of the alkali equal to the amount used in making the bisulphite.

The salt must be kept in tightly-closed bottles in a cool place.

Description.—Colorless prisms or powder, resembling potassium sulphite in most of its properties. Odorless.

It has a cooling, saline, sulphurous taste. Soluble in 4 parts of water at 15°; and in 0.9 part of boiling water. Sparingly soluble in alcohol.

SODIUM TARTRATE.

SODII TARTRAS.

$Na_2C_4H_4O_6.2H_2O=230.$

Tartaric acid	130 parts
Sodium carbonate	251 parts
Distilled water.	

Dissolve the sodium carbonate in the water. Add the tartaric acid gradually, stirring after each addition until dissolved. Filter, evaporate and crystallize, or evaporate the filtrate to dryness to obtain a granular product.

Reaction. $Na_2CO_3+H_2C_4H_4O_6=Na_2C_4H_4O_6+H_2O+CO_2.$

Notes. 148 parts of sodium bicarbonate may be used instead of 251 parts of normal sodium carbonate.

Description.—Colorless, transparent crystals; odorless; of cooling, mildly saline taste. Soluble in 5 parts of water at 15°. Insoluble in alcohol. Neutral to litmus paper.

SODIUM TETRABORATE.

SODII TETRABORAS.

Borax.

$Na_2B_4O_7.10H_2O=381.$

[Sodium Borate. Sodium Pyroborate.]

Borax occurs native in Persia, Thibet, and other western Asiatic countries, and in large deposits in California and Nevada, either alone or with other borates. Most of the borax now used is made

from crude boric acid by fusion with dried sodium carbonate, solution in water, and crystallization.

Description.—Colorless, transparent crystals or white powder, inodorous, having a sweetish, alkaline taste. Slightly efflorescent in warm, dry air. Soluble in 16 parts of water at 15° C. and in one-half its own weight of boiling water. Insoluble in alcohol. Soluble in 1 part of glycerin at 80° C.

Crystallized Sodium Tetraborate.

Borax	1 part
Water	16 parts

Dissolve the borax in the water by the aid of heat, filter the solution, and crystallize the salt by spontaneous evaporation of the solvent in the usual way.

Turbidated Borax.

Borax	1 part
Boiling water	2 parts

Dissolve, filter, and cool the solution rapidly, constantly stirring it, to as low a temperature, above freezing, as may be conveniently attained.

Collect, drain, and dry the salt.

SODIUM THIOSULPHATE.

SODII THIOSULPHAS.

$N_2SO_3S.5H_2O=248.$

["Sodium Hyposulphite."]

This salt, which is a monothiosulphate, may be prepared by boiling sulphur with a solution of sodium hydroxide and then passing sulphur dioxide into the liquid until colorless. The solution is then brought to crystallization.

It may also be made by boiling sulphur with a solution of sodium sulphite.

Commercial sodium thiosulphate may be purified by recrystallization. For this purpose it should be dissolved in its own weight

of distilled water, the solution filtered, and evaporated slowly. Large crystals are easily obtained.

It should be kept in tightly closed bottles.

Description.—Colorless, odorless crystals, soluble in 0.65 part of water at 15° C. and in about 0.5 part at 20° C. Insoluble in alcohol.

["Sodium hyposulphite" is an unfortunate misnomer for real sodium hyposulphite is Na_2SO_2.]

SODIUM VALERATE.

SODII VALERIANAS.

$NaC_5H_9O_2=124.$

Iso-amylic alcohol	1 part
Potassium dichromate	3 parts
Sulphuric acid	4 parts
Water	4 parts
Solution of sodium hydroxide, sufficient.	

Dilute the sulphuric acid with one-half of the water. Dissolve the potassium dichromate in the remainder of the water with the aid of heat. When both liquids are cold mix them with the amylic alcohol in a retort or flask, with occasional brisk agitation until the temperature of the mixture has fallen to about 32° C. Connect with a condenser, and distil until about 4 parts of distillate has been obtained. Saturate the distilled liquid accurately with the solution of sodium hydroxide, remove any oily fluid which floats on the surface, evaporate until watery vapor ceases to escape, and then raise the heat cautiously until the salt is liquefied. When the product has cooled and solidified, break it into pieces, and immediately put it into a stoppered bottle.

Reaction. $3C_5H_{12}O+2K_2Cr_2O_7+8H_2SO_4=$
$3C_5H_{10}O_2+2K_2SO_4+2(Cr_2(SO_4)_3)+11H_2O.$

Notes. The dichromate of potassium is decomposed by the sulphuric acid, the products being potassium sulphate and chromic acid. The amylic alcohol is then oxidized by the chromic acid, and when the mixture is subjected to distillation, the distillate consists of valeric acid and amyl valerate (or apple oil).

When sodium hydrate is added to the liquid, the amyl valerate separates as an oily layer, whilst the valeric acid is neutralized, forming sodium valerate. If the oily amyl valerate be warmed with the soda solution it is decomposed, yielding sodium valerate and amylic alcohol, which also appears as an oily liquid on the surface of the solution. After removing the oily fluid, the solution of sodium valerate is evaporated and the salt fused.

Description.—White crystals, soluble in water and alcohol. The crystals absorb water from moist air, and liquefy. They melt without decomposition, at a temperature of 140°.

STRONTIUM BROMIDE.

STRONTII BROMIDUM.

$SrBr_2.6H_2O=355.5.$

Prepared by saturating hydrobromic acid with pure strontium carbonate, filtering, and evaporating the solution to crystallization. Instead of strontium carbonate the hydroxide may be used. (See Strontium hydroxide, p. 611.) The crystals must be dried cautiously, as the salt effloresces at a higher temperature.

Reaction. $SrCO_3+2HBr=SrBr_2+H_2O+CO_2.$

Strontium bromide may also be prepared from the hydroxide by double decomposition with either ammonium bromide or iron bromide.

Description.—Colorless, transparent crystals; odorless; taste saline, bitterish. Soluble in its own weight of cold water, and in one-half its weight of boiling water. Readily soluble also in alcohol.

STRONTIUM CARBONATE.

STRONTII CARBONAS.

$SrCO_3=147.5.$

Dissolve any desired quantity of commercial strontium nitrate in ten times its own weight of distilled water. Add gradually and during constant stirring a water-solution of potassium dichromate to precipitate any barium present and until a clear yellow liquid is produced. Let the liquid stand for 24 hours; then filter. Add to

the filtrate, heated at a temperature of 100° C., a sufficient quantity of sulphurous acid to cause the disappearance of the characteristic color of the chromic acid and the appearance of a greenish color due to the formation of chromic salt, meanwhile maintaining the temperature at the boiling point of water. Boil the liquid until the excess of sulphurous acid is expelled. Continue the boiling and add enough strontium carbonate to cause the complete precipitation of the chronium by the strontium carbonate. Filter. Pour the filtrate into a filtered solution of sodium carbonate used in excess. Wash the precipitate until the washings are tasteless and leave no perceptible residue on evaporation. Dry the product at a moderate heat.

Description.—An amorphous, white, odorless and tasteless, insoluble powder.

STRONTIUM CHLORIDE.

STRONTII CHLORIDUM.

$SrCl_2.6H_2O=266.3$.

Strontianite, in powder	1000 Gm
Hydrochloric acid (32% of HCl)	1550 Gm
Strontium hydroxide	25 Gm
Water	500 Gm

Mix the powdered strontianite in a porcelain dish with the water. Heat the mixture to about 60°. Stir well. Add the acid in several portions, waiting after each addition until the effervescence has subsided before adding more. When all of the acid has been added heat to boiling for half an hour. Then add the strontium hydroxide and stir well. The solution should be alkaline in reaction; if it is not, add more of the strontium hydroxide to produce an alkaline reaction. Keep the liquid hot for an hour longer, stirring frequently. Then filter. Add enough hydrochloric acid to the filtrate to render it acid. Evaporate to 1.30 sp. w. Set it aside to cool and crystallize. Drain and dry the crystals.

The mother-liquor yields more salt on evaporation in the usual way.

Reaction. $SrCO_3+2HCl=SrCl_2+H_2O+CO_2$.

Notes. The strontium chloride thus obtained is not pure. The principal impurity is barium chloride, for the strontianite contains barium carbonate. Calcium salt is also contained in the product.

It may be partially purified as follows:

Dissolve it in water. Add enough ammonia water to impart an alkaline reaction on litmus paper. Stir well. Filter. Add enough diluted sulphuric acid to the filtrate to render the liquid decidedly acid in reaction. Let stand for two days. Filter. Add enough pure strontium carbonate to neutralize the free acid and leave an excess of strontium carbonate undissolved. Set the mixture in a warm place for a day. Filter again. Evaporate the filtrate tc dryness. Dissolve the residue in three times its weight of water, filter, and evaporate to crystallization.

The barium chloride may also be partially precipitated from the solution of strontium chloride by the addition of alcohol in which strontium chloride is soluble while the barium chloride is insoluble in it. Or the strontium chloride may be dissolved in alcohol, the solution filtered and evaporated to dryness, the salt being then recrystallized from a water solution.

Description.—Long, colorless, deliquescent crystals, readily soluble in water and in alcohol.

STRONTIUM HYDROXIDE.

STRONTII HYDROXIDUM.

$Sr(OH)_2.8H_2O=265.50.$

Add 8 parts of water to 5 parts of the oxide. A solid crystalline mass is formed. Dissolve this in 40 parts of boiling water; filter hot; let the filtrate cool slowly. Collect and dry the crystals.

Description.—Transparent, colorless, deliquescent crystals.

STRONTIUM IODIDE.

STRONTII IODIDUM.

$SrI_2.6H_2O=448.5.$

May be prepared from iron iodide and strontium hydroxide (see Calcium Iodide, p. 326).

Description.—Colorless, transparent plates; odorless; taste saline, bitterish. Deliquescent. Becomes yellow on exposure to air and light. Soluble in 0.6 part of water at 15°, and in 0.27 part of boiling water. Also soluble in alcohol.

Must be kept in tightly-closed bottles, protected against light

STRONTIUM LACTATE.

STRONTII LACTAS.

$Sr(C_3H_5O_3)_2.3H_2O=319.$

Strontium carbonate ..... 1 part
Lactic acid, sufficient.

Place the strontium carbonate together with 5 parts of distilled water in a porcelain dish over a water-bath. Add the lactic acid gradually, stirring constantly, until but little of the strontium carbonate remains undissolved. Continue heating the mixture at the maximum heat of the bath, until the lactic acid it saturated, adding, if necessary, a little more strontium carbonate so as to have a sufficient excess to insure complete neutralization. Let the liquid cool, filter it, and evaporate it at a temperature of between 60° and 80° C. until a crystalline mass is formed on cooling. Dry the product in a current of air without the aid of heat.

Keep it in well-stoppered bottles.

Description.—A white, granular powder; odorless; taste saline, slightly bitter. Soluble in 4 parts of water at 15°; in less than one-half its weight of boiling water. Also soluble in alcohol.

STRONTIUM NITRATE.

STRONTII NITRAS.

$Sr(NO_3)_2=211.5.$

Strontianite, in powder	200 parts
Nitric acid (68% of HNO_3)	250 parts
Strontium hydroxide	5 parts
Water	300 parts

Mix the strontianite with the water. Warm the creamy mixture. Add the nitric acid gradually, stirring well and taking care not to allow the liquid to run over from a too violent effervescence. When all of the acid has been added, heat the liquid to expel the carbon dioxide. Add the strontium hydroxide, stir well, and digest for an hour, stirring frequently. Filter. Acidify the filtrate with nitric acid. Evaporate nearly to dryness, and place the wet crystalline mass of salt in a funnel to drain. Dry the product with the aid of moderate heat.

Reaction. $SrCO_3+2HNO_3=Sr(NO_3)_2+H_2O+CO_2$.

Description.—Transparent, anhydrous crystals, readily soluble in water.

STRONTIUM OXIDE.

STRONTII OXIDUM.

$SrO=103.5$.

Prepared by strongly heating anhydrous strontium nitrate to redness for an hour or two.

Reaction. $2Sr(NO_3)_2+2SrO+2N_2O_4+O_2$.

Description.—A dirty-white porous solid, or a white, amorphous, odorless powder of caustic, alkaline taste.

Must be kept in tightly-closed bottles.

SULPHUR.

Sublimed sulphur contains usually some arsenic and free sulphuric acid. It is purified as follows:

Washed Sulphur.

SULPHUR LOTUM.

Sublimed sulphur	100 Gm
Ammonia water	10 ml
Water, sufficient.	

Add the sulphur to 100 ml of water previously mixed with the

ammonia water, and digest for three days, agitating occasionally. Then add 100 ml of water; transfer the mixture to a muslin strainer, and wash the sulphur with water until the washings cease to give an alkaline reaction on test-paper. Then drain, press strongly, dry it at not over 40° C. (104° F.), and powder by running it through a No. 30 sieve.

Notes. The object of this process is to neutralize and wash away the sulphuric acid which is always present with sublimed sulphur, and the digestion for three days is intended to remove also any arsenic present. To effect the extraction of the arsenic, continued digestion is required, and three days is not too long, as the ammonia is very diluted. Care should be taken, however, not to use higher heat than 40° C. (104° F.), because above that temperature a good deal of ammonium hydrosulphide may be formed, and at the boiling point the sulphur will dissolve as long as sufficient ammonia remains to act upon it. Boiling water is, therefore, not to be used. It is under all circumstances necessary to wash out all the ammonia and ammonium salt from the sulphur before drying it.

It is important to dry the washed sulphur thoroughly before putting it in its receptacle, for the presence of any moisture would result in the formation of sulphuric acid again, rendering a repetition of the washing necessary.

Reaction. $As_2S_3+H_2SO_4+6H_4NOH=$

$$(H_4N)_2SO_4+(H_4N)_2HAsO_3+(H_4N)_2HAsS_3+3H_2O.$$

Description.—A fine, yellow, odorless and tasteless powder. Insoluble in water and in official alcohol.

Precipitated Sulphur.

SULPHUR PRAECIPITATUM.

Sublimed sulphur	2 parts
Lime	1 part
Official hydrochloric acid, water,	each sufficient.

Slake the lime and make it into a uniform mixture with 10 parts of water. Add the sulphur, previously well dried and sifted, mix well, add 25 parts of water, and heat the mixture to boiling for

one hour, stirring constantly, and replacing the water lost by evaporation. Then cover the vessel, allow the contents to cool, pour off the clear solution, filter the remainder, and unite the liquids.

Add gradually hydrochloric acid previously diluted with an equal volume of water, until the liquid is nearly neutralized, being careful to leave it still distinctly alkaline in reaction and of yellow color.

Collect the precipitate on a strainer, and wash it with water until the washings are tasteless and no longer give an acid reaction on test-paper. Then dry it at a gentle heat.

Reactions. The lime is first slaked, $CaO+H_2O=Ca(OH)_2$. Then, when sulphur is boiled with the calcium hydroxide, the following reaction occurs: $3Ca(OH)_2+12S=2CaSS_4+CaS_2O_3+3H_2O$. The solution, containing calcium tetrathiosulphate and calcium thiosulphate, is next treated with hydrochloric acid, when sulphur is precipitated: $CaSS_4+2HCl=CaCl_2+4S+H_2S$.

Notes. The quantity of lime used is purposely somewhat in excess of the amount required by theory, to make up for carbonate contained in it. A larger excess would, however, result in loss because the compounds then formed would yield larger quantities of H_2S when the hydrochloric acid is added.

By long boiling the calcium mono-thiosulphate is split up into sulphite and sulphur. A portion of the sulphite is oxidized to sulphate, which is precipitated.

If the first portion of hydrochloric acid is added very slowly no hydrogen sulphide is formed, because the reaction then is: $2CaSS_4+2HCl=CaCl_2+Ca(SH)_2+4S_2$. When more acid is added, however, the reaction just described is followed by: $Ca(SH)_2+2HCl=CaCl_2+2H_2S$.

The directions of the Pharmacopœia are to only "nearly" neutralize the solution, and to be careful to leave it slightly *alkaline*. The object of this precaution is to prevent the precipitation of any arsenic present. The calcium thiosulphate is not decomposed unless enough hydrochloric acid be added to impart an acid reaction to the liquid. Hence the sulphur which enters into the structure of the calcium thiosulphate is lost. But if enough hydrochloric acid be added to decompose the thiosulphate ($2CaSS_4+CaSO_3S+6HCl=3CaCl_2+12S_2+3H_2O$), the arsenic will also

be precipitated as follows: $Ca_3(AsS_4)_2+6HCl=As_2S_3+3H_2S+3CaCl_2+2S$.

When the liquid is alkaline, however, all of the arsenic remains in solution.

Care should be taken to arrange so that the hydrogen sulphide gas is led off through a flue, or the operation should be carried out in the open air. The gas is evolved in large enough quantities to be very annoying to the operator.

The acid must be added to the solution of the sulphur salts and not *vice versa;* moreover, the acid must be diluted as directed; otherwise an oily product (H_2S_2?) is formed which afterwards is difficult to eliminate and which but slowly decomposes, giving off H_2S.

The well-washed precipitated sulphur must be rapidly but thoroughly dried at a moderate heat and kept in tightly-closed bottles.

Description.—An odorless and tasteless, pale-yellowish, fine powder, insoluble in water; readily soluble in carbon disulphide; also soluble in ether, chloroform, fixed oils and volatile oils, benzin, and benzol. Melts at 115° C., and at a higher heat volatilizes without residue, or burns in the air forming sulphur dioxide.

SULPHUR DIOXIDE.

SULPHURIS DIOXIDUM.

$SO_2=64$.

Copper turnings or scraps	100 Gm
Concentrated sulphuric acid	300 Gm
Water	100 Gm

Put the sulphuric acid in a porcelain dish of about one liter's capacity; set the acid into rapid whirling motion by circulatory stirring in one direction with a glass rod. Then *slowly* pour the water into the acid, continuing the stirring uninterruptedly until all the water has been added.

Put the copper into a round-bottomed flask of about one and one-half liter's capacity. Provide the flask with a twice perforated rubber stopper carrying a thistle tube and a delivery tube. Connect the delivery with a wash-bottle of about one-half liter's

capacity containing about 200 Cc. of water and provided with a thrice perforated rubber stopper carrying a thistle tube and second delivery tube with connections by means of which the sulphur dioxide may be conducted into any desired receiver.

Add the mixture of sulphuric acid and water to the copper, and, having inserted the stoppers and fittings and completed all connections, heat the flask until the reaction begins. Then remove the burner from under the flask so long as the reaction continues briskly, applying heat again only when the evolution of sulphur dioxide becomes too slow.

Reaction. $Cu+2H_2SO_4=CuSO_4+2H_2O+SO_2$.

Notes. It will be seen that a large excess of copper is prescribed; this is to facilitate the evolution of the gas. The heat generated by the reaction itself, after it has been started by the application of heat from without, is sufficient to maintain the action for some time. A strong dish should be placed under the flask while the reaction proceeds without the use of the burner, so that, if the flask should break, its contents may be caught in the dish.

When the acid is saturated the copper sulphate formed in the flask may be dissolved in water and recovered by crystallization.

The amount of sulphuric acid ordered will furnish, when entirely consumed, about 90 to 96 Gm of SO_2.

Sulphur dioxide may be conveniently prepared in this way when required for making sulphurous acid and water-soluble sulphites, and for other purposes. Larger quantities of the materials and apparatus of greater capacity are, of course, to be employed when requisite.

Sulphur dioxide may also be prepared by heating sulphuric acid with charcoal, and this method is prescribed by the Pharmacopœia for the preparation of sulphurous acid. It is economical, because all of the sulphuric acid decomposed yields SO_2, whereas the copper process involves the formation of copper sulphate which consumes one-half of the sulphuric acid decomposed. If the copper sulphate is recovered, however, the difference in cost is insignificant. But the method of generating sulphur dioxide by reducing sulphuric acid by heating this with carbon can not be employed in cases where the carbon dioxide formed in that process is objectionable.

SULPHUR IODIDE.

SULPHURIS IODIDUM.

Consists chiefly of S_2I_2=317.

Washed sulphur	1 part
Iodine	4 parts

Mix them thoroughly by trituration in a glazed porcelain mortar (or in a glass mortar). Put the mixture into a flask, beaker or wide-mouthed bottle, close the top loosely, and apply gentle heat (not exceeding 60° C.) by means of a water-bath until the elements combine, which is known by the fact that the mass assumes a uniformly dark color. Then raise the temperature until the mass becomes liquid. Pour the fused sulphur iodide upon a porcelain tile or plate or upon any other convenient cold surface not affected by it. Break the cake, when cool, into small pieces and transfer the product to small glass-stoppered bottles.

Must be kept in a cool place.

Notes. The sulphur and iodine, when previously, intimately mixed, combine easily. The compound S_2I_2 is, however, not stable. If, during the process of heating the iodine and sulphur together, or in the fusion of the iodide, any portion of the iodine should become volatilized and condense upon the sides or in the upper part of the container in which the reaction is performed, this iodine must be returned to the liquid portion by bringing it in contact with the fused mass, which is easily accomplished by inclining the container in the direction required. Full water-bath heat is sufficient to effect the liquefaction of the product. The flask or other vessel in which the iodide of sulphur is made may be covered with a watch crystal during the operation.

Description.—A brittle, grayish-black, crystalline solid of somewhat metallic lustre, having an odor of iodine and an acrid taste. Insoluble in water; soluble in 60 parts of glycerin. Decomposed by alcohol and ether, which dissolve the iodine, leaving the sulphur. It loses iodine on exposure. Decomposes when heated long or to a high temperature; but it should leave no residue on volatilization.

TIN CHLORIDE.

STANNI DICHLORIDUM.

(Stannous Chloride. Tin Salt.)

$SnCl_2.2H_2O$=225.8.

Tin, granulated	100 parts
Hydrochloric acid (35% of HCl)	175 parts

Dissolve the tin in the hydrochloric acid by digestion. Finally heat at about 60° until all action ceases and the acid is saturated. Let cool and settle. Decant the clear solution and evaporate it to the sp. w. of 1.985. Then set it aside in a covered dish to crystallize. Drain the crystals well in a covered funnel, and dry the product in a desiccator over sulphuric acid. Bottle the salt as soon as dry.

Reaction. $Sn+2HCl=SnCl_2+H_2$.

Description.—Colorless crystals, soluble in 0.37 part of water at 15°. Decomposed by more water.

ZINC ACETATE.

ZINCI ACETAS.

$Zn(C_2H_3O_2)_2.2H_2O$=219.3.

Zinc oxide	300 Gm
Acetic acid	1000 ml
Distilled water	1000 ml

Mix 600 ml of the acid with the whole of the water in a flask, add the zinc oxide, agitate gently, and warm the mixture to about 60° C.; add the remainder of the acid gradually, continuing the digestion until no more of the oxide is dissolved. Heat the solution to the boiling point; filter while hot, add a little acetic acid (about 10 ml) to insure that the acid is present in slight excess. Set the solution in a cool place for two days to crystallize. Decant the mother-liquor, evaporate it over a water-bath to one-half, acidulate with acetic acid as before, if necessary, and again set

aside to crystallize in a cool place. Put the crystals in a funnel to drain; then dry them on filter paper or on a porous tile, without the aid of heat.

Must be kept in a tightly-corked bottle.

Reaction. $ZnO+2HC_2H_3O_2=Zn(C_2H_3O_2)_2+H_2O$.

Notes. The same result will be obtained if 420 Gm of zinc carbonate be used instead of 300 Gm of the oxide. The carbonate should be added gradually on account of the effervescence caused by the liberation of carbonic acid, and the solution formed is then to be boiled a few minutes to expel the carbonic acid perfectly.

When the preparation is made on a manufacturing scale from commercial zinc oxide or carbonate, it is necessary to digest the solution of acetate of zinc for several days with some pure metallic zinc, or with pure zinc oxide, in order to remove any lead, copper, cadmium and iron that may be present. By this digestion, however, some basic zinc acetate is formed, which must be converted into normal acetate by the addition of more acetic acid before crystallizing. A solution of basic salt will not crystallize, but the presence of a little free acetic acid facilitates the crystallization.

When the first crop of crystals has been collected, the mother-liquor must be evaporated at a temperature not above 80° C. (176° F.) until a pellicle forms, after which it is set aside in a cool place. If the evaporation is carried too far a precipitate of zinc acetate containing but one molecule of water of crystallization will deposit.

Zinc acetate may also be made by double decomposition from 38 parts of lead acetate and 29 parts of zinc sulphate, each dissolved in 80 parts of distilled water, the solutions being filtered and then mixed. The reaction is then:

$$Pb(C_2H_3O_2)_2+ZnSO_4=Zn(C_2H_3O_2)_2+PbSO_4.$$

Description.—Soft, white, pearly crystals, of a faintly acetous odor and an astringent, afterwards metallic taste. Soluble in 2.7 parts of water and in 36 parts of alcohol at 15°; in 1.5 parts of boiling water, and in 3 parts of boiling alcohol. Reaction acid.

ZINC BROMIDE.

ZINCI BROMIDUM.

$ZnBr_2$=225.3.

Zinc sulphate	8 parts
Potassium bromide	7 parts
Boiling distilled water, sufficient.	

Dissolve the sulphate in 10 parts and the bromide in 6 parts of the water. Mix the solutions while hot. When cool add 40 parts of alcohol, and filter the mixture through asbestos. Evaporate the filtrate to dryness, and granulate the product.

Reaction. $ZnSO_4+2KBr=K_2SO_4+ZnBr_2$.

Zinc bromide may also be made from zinc sulphate and barium bromide.

Another Method.

Zinc	66 parts
Bromine	160 parts
Water	250 parts

Put the zinc and water in a flask of about 500 Cc. capacity. Add the bromine in small portions at a time, waiting after each addition until the red color has nearly disappeared and the reaction subsided. When all of the bromine has been added, filter the liquid and evaporate to dryness.

Description.—A white, granular salt; odorless; of sharp, saline, metallic taste Very hygroscopic. Readily soluble in water and in alcohol.

ZINC CARBONATE; PRECIPITATED.

ZINCI CARBONAS PRAECIPITATUS.

$2ZnCO_3.3Zn(OH)_2$=548.5.

Zinc sulphate	20 parts
Sodium carbonate	21 parts
Boiling water, sufficient.	

Dissolve the sodium carbonate in 40 parts of the water in a porcelain capsule. Dissolve the zinc sulphate in an equal quantity of boiling water. Place the capsule containing the solution of sodium carbonate on a sand-bath so as to keep the solution hot; then gradually add the solution of zinc sulphate, stirring briskly. Boil the mixture for fifteen minutes after the effervescence has ceased; then let the precipitate subside. Decant the mother-liquor and throw it away. Add 120 parts of boiling water to the precipitate, and stir well, let settle once more, and repeat the washing by affusion and decantation of hot distilled water until the washings cease to give any precipitate with test solution of barium chloride. Collect the precipitate on a muslin strainer or on a paper filter, let it drain thoroughly, and then dry it at a gentle heat.

Reaction.

$$5Na_2CO_3+5ZnSO_4+3H_2O$$
$$=2ZnCO_3.3Zn(OH)_2+5Na_2SO_4+3CO_2.$$

Notes. The solutions are used hot in order to expel all the carbonic acid which is set free, carbon dioxide escaping with effervescence. If cold solutions are used a portion of the precipitate redissolves in the carbonic acid held in the liquid. The zinc sulphate is to be added to the sodium carbonate in order to keep the latter in excess, and by having the soda solution hot, the further advantage is gained that the precipitate is more readily washed free from sodium salt.

Description.—A fine, white, odorless and tasteless, insoluble powder.

ZINC CHLORIDE.

ZINCI CHLORIDUM.

$$ZnCl_2=136.1.$$

A solution of zinc chloride is prepared as described in the article below. It is then evaporated to dryness and the residue fused, or the product is granulated. It must be bottled while

warm in warm dry bottles, and should be kept in small, tightly-stoppered bottles.

Description.—Zinc chloride should be perfectly white. It is very deliquescent. Its solution is strongly acrid, corrosive, and of acid reaction.

Zinc Chloride Solution.

LIQUOR ZINCI CHLORIDI; U. S.

The official solution of zinc chloride (U. S. P., 1890) contains about 50 per cent of $ZnCl_2$. It is prepared as follows:

Granulated zinc	240 Gm
Hydrochloric acid	840 Gm
Nitric acid	12 Gm
Precipitated zinc carbonate	12 Gm
Distilled water, sufficient.	

Put the zinc in a glass or porcelain vessel, and add 150 ml of distilled water. Then add the hydrochloric acid gradually. Digest until the acid shall have become saturated. Decant the solution from the undissolved excess of zinc.

Add the nitric acid to the solution, evaporate the mixture to dryness and heat the dry mass to fusion at a temperature not exceeding 115° C.

Let the fused zinc chloride cool and then dissolve it in a sufficient quantity of distilled water to make the weight of the solution one thousand grams.

Add the precipitated zinc carbonate, agitate the mixture occasionally during twenty-four hours, and then set it aside, at rest, until clarified by subsidence.

Finally separate the clear solution by decantation or by means of a glass syphon, and keep it in glass-stoppered bottles.

Reaction. $Zn+2HCl=ZnCl_2+H_2$.

Notes. The metal dissolves in the hydrochloric acid with the liberation of heat and the evolution of hydrogen. Instead of adding the acid to the zinc, the metal may be added to the acid.

The effervescence caused by the escape of the hydrogen may be very active at first, but subsides as the zinc chloride increases and the acid diminishes. When but little acid remains digestion

is necessary to cause complete saturation, and further digestion is desirable to cause the separation of certain foreign metals which may have been contained in the zinc.

Commercial zinc is usually contaminated with one or more of the metals, copper, lead, arsenic, cadmium, iron and manganese. When an excess of zinc is used, so that a portion of it remains undissolved at the end of the digestion, several of these other metals dissolved together with the zinc are precipitated by the latter on prolonged digestion.

But one of the most common impurities is iron, which can not be precipitated by digestion with an excess of zinc. To remove the iron (and manganese) nitric acid is added to convert the ferrous chloride to ferric, and the solution is evaporated to dryness and the residue fused to expel the excess of nitric acid. The fused zinc chloride (containing any iron chloride derived from the iron in the zinc used) is then redissolved and the solution shaken with precipitated zinc carbonate, which removes the iron which is precipitated in the form of hydroxide.

Solution of zinc chloride cannot be filtered through paper, because the latter swells until its pores are closed and is then corroded. It may be filtered through powdered and washed glass, or through purified asbestos. Decantation is sometimes sufficient. A dilute solution may be filtered through paper, but the organic substances then taken up by it blacken the product as soon as sufficiently concentrated by evaporation.

Description.—A clear, colorless, odorless liquid of astringent, sweetish, metallic taste and acid reaction. Corrosive in its action upon organic matter. Sp. w. about 1.535 at 15°.

ZINC CYANIDE.

ZINCI CYANIDUM.

$Zn(CN)_2 = 117.3$.

Prepared by precipitating a solution of zinc acetate with hydrocyanic acid.

Description.—A colorless salt, insoluble in water and in alcohol, but soluble in solutions of alkalies with which it forms water-soluble double salts.

ZINC IODIDE.

ZINCI IODIDUM.

ZnI_2=318.3.

Granulated zinc	3 parts
Iodine	10 parts
Distilled water	20 parts

Digest together in a flask until colorless and free from the odor of iodine. Filter through asbestos or powdered glass, and evaporate the filtrate rapidly to dryness at a moderate heat.

Must be kept in small well-closed vials.

Description.—A white, granular salt; odorless; taste sharp, saline, metallic. Very hygroscopic. Readily soluble in water, alcohol and ether.

ZINC LACTATE.

ZINCI LACTAS.

$Zn(C_3H_5O_3)_2.3H_2O$=297.3.

Prepared by neutralizing warm dilute lactic acid with zinc carbonate, and crystallizing.

Can also be prepared by double decomposition between sodium lactate and zinc chloride.

Description.—White or colorless quadrangular crystals, inodorous, and having an acidulous metallic taste, and acid reaction. Soluble in 58 parts of cold and 6 parts of boiling water; nearly insoluble in alcohol.

ZINC OLEATE.

ZINCI OLEAS.

$Zn(C_{18}H_{33}O_2)_2$=627.3.

Zinc acetate	2 parts
Powdered white castile soap	5 parts

Dissolve the zinc acetate in 300 parts of water, and the soap in

200 parts of hot water. Add the cold soap solution to the solution of zinc acetate, stirring briskly. Drain the precipitated zinc oleate on a muslin strainer, and wash it several times with cold water. Dry it on paper or muslin, without the aid of heat.

Reaction. $2NaC_{18}H_{33}O_2+Zn(C_2H_3O_2)_2=Zn(C_{18}H_{33}O_2)_2+2NaC_2H_3O_2$.

Notes. The sodium acetate is easily washed away from the precipitated oleate. The yield is about 2.4 parts.

Description.—A soft, white powder, having a soapy feel.

Another Method.

Zinc sulphate	3 parts
Powdered Castile soap	5 parts

Dissolve the zinc sulphate in 300 parts of water, and the soap in 200 parts of hot water. Keep the soap solution at the temperature of about 40°, and add to it slowly and with brisk stirring, the solution of zinc sulphate. Collect the precipitate on a muslin strainer, wash it with water of 40° temperature, drain it well, and dry it at not over 40°. Pass the dried friable mass through a sieve.

Official Zinc Oleate.

Zinc oxide	5 parts
Oleic acid	95 parts

Put the oleic acid in a roomy porcelain dish, and sift the zinc oxide into the acid, stirring well so as to mix the two thoroughly and avoid the formation of lumps. Let the mixture stand several hours, stirring occasionally. Then heat the mixture over a water-bath until the oxide is completely dissolved.

Reaction. $ZnO+2HC_{18}H_{33}O_2=Zn(C_{18}H_{33}O_2)_2+H_2O$.

Notes. If the quantities operated upon are small, the zinc oxide,

previously sifted, may well be mixed with the oleic acid by trituration in a mortar, the acid being gradually added to the oxide. The mixture should be allowed to stand at least two hours before it is heated.

It will be observed that a very large excess of oleic acid is used, so that the preparation is a mixture of zinc oleate and oleic acid.

The zinc oxide used must be quite free from iron; otherwise the product will be pinkish, or reddish, or yellowish, according to the condition and amount of iron in it.

Description.—A soft, white, homogeneous ointment is formed out of zinc oxide and oleic acid when the percentage of zinc oxide is sufficient; with less zinc oxide the product is fluid or semi-fluid. True zinc oleate, without an excess of oleic acid, is a powder. The official zinc oleate, obtained by the formula here given, is a soft ointment.

Pure zinc oleate is prepared as described under Zinci Oleas.

ZINC OXIDE.

ZINCI OXIDUM.

$ZnO=81.3$.

Calcine the official zinc carbonate in a dish at a low red heat until a cooled sample, mixed with a little water, no longer effervesces with diluted hydrochloric acid.

Reaction. $2(ZnCO_3).3Zn(OH)_2=5ZnO+3H_2O+2CO_2$.

Notes. Too high heat renders the product yellow, and this yellowish tint may be retained for a long time. [All pure zinc oxide turns yellow when heated but becomes white again on cooling unless it was heated too strongly. A zinc oxide containing much iron is always discolored even if not overheated in the process of calcination.] Overheating also makes zinc oxide less soft to the feel.

ZINC PHENOLSULPHONATE.

ZINCI PHENOLSULPHONAS.

$Zn(C_6H_5SO_4)_2.8H_2O=555.3.$

(Sulphocarbolate of Zinc.)

Crystallized phenol	20 parts
Sulphuric acid	25 parts
Barium carbonate	40 parts
Zinc sulphate	29 parts
Water.	

Add the phenol to the acid, mix well, and heat the mixture at 55° in a porcelain dish over a water-bath for several days. Add 400 parts of water and mix well. Then neutralize by adding the barium carbonate, stirring the whole thoroughly. Filter. Reject the precipitate. To filtrate add the zinc sulphate previously dissolved in 200 parts of water. Now filter away the precipitated barium sulphate, slightly acidulate the filtrate with sulphuric acid, and evaporate to crystallization.

Purify the product by repeated re-crystallizations.

Reactions. $2HC_6H_5SO_4+BaCO_3=Ba(C_6H_5SO_4)_2+H_2O+CO_2$; and then, $Ba(C_6H_5SO_4)_2+ZnSO_4=Zn(C_6H_5SO_4)_2+BaSO_4$.

Notes. The solution of zinc sulphate should be added to the solution of sulpho-carbolate of barium until exactly precipitated—that is, the further addition of zinc sulphate must be discontinued as soon as it no longer causes any further precipitation. Sulpho-carbolate of zinc crystallizes more readily, and without reddish color, from a solution acidulated with sulphuric acid.

Description.—Colorless crystals, readily soluble in water and in alcohol.

ZINC SALICYLATE.

ZINCI SALICYLAS.

$$Zn(C_7H_5O_3)_2.3H_2O=393.3.$$

Zinc oxide	3 parts
Salicylic acid	10 parts
Distilled water, sufficient.	

Mix the salicylic acid with 50 parts of water, and heat the mixture. Add gradually the zinc oxide previously mixed with 15 parts of water, until no more dissolves. Filter, and set aside to crystallize.

Description.—Colorless crystals, soluble in water and in alcohol.

ZINC SULPHATE.

ZINCI SULPHAS.

$$ZnH_2SO_5.6H_2O=287.3.$$

Zinc clippings, or granulated zinc	4 parts
Sulphuric acid	5 parts

Dilute the acid with 30 parts of water; add the zinc gradually. When effervescence has ceased, digest with the undissolved portion of the metal for a few days. Filter. Evaporate the filtrate until a drop becomes turbid on cooling. Then let the solution become cold, stirring constantly so that small crystals may be formed. Collect and dry these on muslin. Evaporate the mother-liquor to obtain more crystals.

Reaction. $Zn+H_2SO_4+7H_2O=ZnH_2SO_5.6H_2O+H_2$.

Notes. The zinc should be nearly free from other metals. If it contains iron, the zinc sulphate contaminated with ferrous sulphate will become yellow in time by the oxidation of the ferrous to ferric salt. Zinc sulphate containing iron will, of course, be unfit for the preparation of zinc oxide.

Any iron and manganese present in the metal will not be re-

moved by digestion with an excess of zinc; but lead, copper, and cadmium, if present, are thus separated. The gray flocculi which are formed when the zinc is dissolved in the acid are mainly lead.

Pure zinc dissolves with difficulty in the acid; if it contains iron it dissolves more rapidly in proportion to the iron in it.

Iron is removed as described below in the recrystallization of zinc sulphate.

The solution should not be too concentrated for crystallizing, for when the salt is crystallized from a warm solution (over 30° C.) the crystals formed contain less water of crystallization ($ZnSO_4.5H_2O$). The crystals should be dried without the aid of heat, or at least at a temperature not exceeding 30° C.

Impure zinc sulphate may be purified to a great extent by recrystallization. The salt crystallizes best from a slightly acid solution.

Zinc sulphate may also be made by saturating sulphuric acid with zinc oxide and crystallizing.

If large crystals are desired let the saturated solution be evaporated slowly at the ordinary temperature and without disturbing it.

Description.—Colorless, transparent crystals; odorless; taste metallic, astringent. Soluble in 0.6 part of water at 15°, and in 0.2 part of boiling water; also in 3 parts of glycerin. Insoluble in alcohol. Reaction acid.

Recrystallized Zinc Sulphate.

White vitriol	5 parts
Water	3 parts

Make a solution. Acidify it with 1 part of diluted sulphuric acid. Boil it for a few minutes. Filter. Conduct a current of chlorine into the warm solution until it acquires the odor of it. Add a small quantity of iron-free zinc oxide or zinc carbonate to produce a slightly turbid mixture containing but little undissolved zinc compound. Digest for a day or two, shaking occasionally. Bring it to the boiling point. Filter. Acidify again with diluted sulphuric acid. Crystallize in the usual way.

Notes. White vitriol contains iron, and consists of an opaque, granular, more or less dirty salt mass. When its solution is

strongly acidified and boiled it can be filtered quite clear. Chlorine converts the iron present into ferric sulphate. The iron is then easily precipitated by digestion with zinc oxide or zinc carbonate in slight excess. The separation of the iron is facilitated by boiling. After filtration the solution is again to be acidified by the addition of about 1 part of diluted sulphuric acid because the salt crystallizes most satisfactorily from an acid solution.

If copper, lead, arsenic, cadmium, or manganese be present, these metals are removed as well as iron by this method.

ZINC SULPHITE.

ZINCI SULPHIS.

$ZnSO_3.2H_2O=179.3.$

Zinc sulphate	60 parts
Sodium sulphite	53 parts
Distilled water, sufficient.	

Dissolve each salt separately in 200 parts of cold distilled water and filter the solutions. Add the zinc salt solution to the solution of the sodium sulphite, stirring well. A precipitate of zinc sulphite will be formed in the course of from twenty to thirty minutes.

Decant the mother-liquor. Wash the precipitate with a limited amount of cold water until the washings are nearly tasteless and no longer contain sodium sulphate. Dry the product without the aid of heat.

Reaction. $ZnSO_4+Na_2SO_3=ZnSO_3+Na_2SO_4.$

Notes. Cold solutions are used and the product washed and dried at the ordinary room temperature because zinc sulphite is liable to be decomposed at a higher temperature, basic salt being formed.

Description.—A white, crystalline powder, practically insoluble in water.

ZINC VALERATE.

ZINC VALERIANAS.

$$Zn(C_5HgO_2)_2.2H_2O=303.3.$$

Sodium valerate	25 parts
Zinc sulphate	29 parts
Distilled water, sufficient.	

Dissolve the salts, separately, each in 200 parts of water; heat the solutions to boiling; mix while hot; let the mixture cool. Collect the crystals, wash them hastily with cold water, and dry them between filter-paper.

Another crop may be had by evaporating the mother liquor to 20 parts, cooling, and separating the crystals formed.

Reaction. $2NaC_5H_9O_2+ZnSO_4=Zn(C_5H_9O_2)_2+Na_2SO_4$.

Notes. As the zinc valerate is lighter than the solution of sodium sulphate, the crystals rise to the surface as they are formed.

The salt may also be made by dissolving 3 parts of zinc oxide in a mixture of 5 parts of valeric acid, 200 parts of alcohol, and 200 parts of water; but the crystallization is tedious on account of the necessity of using but moderate heat in evaporating the solution.

The crystals must be dried without the aid of heat.

Description.—Soft, glistening white crystals, soluble in about 100 parts of water and in 40 parts of alcohol. Taste sweetish astringent, finally metallic.

TABLES.

TABLE OF ATOMIC WEIGHTS.

Aluminum	27.	Lead	206.5
Antimony	120.	Lithium	7.
Arsenic	75.	Magnesium	24.2
Barium	137.	Manganese	55.
Bismuth	208.	Mercury	200.
Boron	11.	Nickel	58.5
Bromine	80.	Nitrogen	14.
Cadnium	112.	Oxygen	16.
Calcium	40.	Phosphorus	31.
Carbon	12.	Platinum	194.
Cerium	139.	Potassium	39.
Chlorine	35.4	Silicon	28.3
Chromium	52.	Silver	108.
Cobalt	59.	Sodium	23.
Copper	63.5	Strontium	87.5
Fluorine	19.	Sulphur	32.
Gold	197.	Tin	119.
Hydrogen	1.	Tungsten	184.
Iodine	126.5	Zinc	65.3
Iron	56.		

MULTIPLES OF THE ATOMIC WEIGHTS OF COMMON ELEMENTS AND ATOMIC GROUPS.

1	H	1	2	3	4	5	6	7	8	9	10	H	1
2	Ag	108	216	324	432	540	648	756	864	972	1080	Ag	2
3	Al	27	54	81	108	135	162	189	216	243	270	Al	3
4	As	75	150	225	300	375	450	525	600	675	750	As	4
5	Ba	137	274	411	548	685	822	959	1096	1233	1370	Ba	5
6	Br	80	160	240	320	400	480	560	640	720	800	Br	6
7	C	12	24	36	48	60	72	84	96	108	120	C	7
8	$C_2H_3O_2$	59	118	177	236	295	354	413	472	531	590	$C_2H_3O_2$	8
9	$C_4H_4O_6$	148	296	444	592	640	788	936	1184	1332	1480	$C_4H_4O_6$	9
10	$C_6H_5O_7$	189	378	567	756	945	1134	1323	1512	1701	1890	$C_6H_5O_7$	10
11	CN	26	52	78	104	130	156	182	208	234	260	CN	11
12	CO_3	60	120	180	240	300	360	420	480	540	600	CO_3	12
13	C_2O_4	88	176	264	352	440	528	616	704	792	880	C_2O_4	13
14	Ca	40	80	120	160	200	240	280	320	360	400	Ca	14
15	Cl	35.4	70.8	106.2	141.6	177.0	212.4	247.8	283.2	318.6	354.0	Cl	15
16	Cu	63.5	127.0	190.5	254.0	317.5	381.0	444.5	508.0	571.5	635.0	Cu	16
17	Fe	56	112	168	224	280	336	392	448	504	560	Fe	17
18	HO	17	34	51	68	85	102	119	136	153	170	HO	18
19	H_2O	18	36	54	72	90	108	126	144	162	180	H_2O	19
20	H_2N	16	32	48	64	80	96	112	128	144	160	H_2N	20
21	H_3N	17	34	51	68	85	102	119	136	153	170	H_3N	21

22	H_4N	18	36	54	72	90	108	126	144	162	180	H_4N	22
23	Hg	200	400	600	800	1000	1200	1400	1600	1800	2000	Hg	23
24	I	126.5	253.0	379.5	506.0	632.5	759.0	885.5	1012.0	1138.5	1265.0	I	24
25	K	39	78	117	156	195	234	273	312	351	390	K	25
26	Li	7	14	21	28	35	42	49	56	63	70	Li	26
27	Mg	24.2	48.4	72.6	96.8	121	145.2	169.4	193.6	217.8	242.0	Mg	27
28	N	14	28	42	56	70	84	98	112	126	140	N	28
29	NO_2	46	93	138	184	230	276	322	368	414	460	NO_2	29
30	NO_3	62	124	186	248	310	372	434	496	558	620	NO_3	30
31	Na	23	46	69	92	115	138	161	184	207	230	Na	31
32	O	16	32	48	64	80	96	112	128	144	160	O	32
33	OH	17	34	51	68	85	102	119	136	153	170	OH	33
34	P	31	62	93	124	155	186	217	248	279	310	P	34
35	PO_2H_2	65	130	195	260	325	390	455	520	585	650	PO_2H_2	35
36	PO_4	95	190	285	380	475	570	665	760	855	950	PO_4	36
37	Pb	206.5	413.0	619.5	826.0	1032.5	1239.0	1445.5	1652.0	1858.5	2065.0	Pb	37
38	S	32	64	96	128	160	192	224	256	288	320	S	38
39	SO_3	80	160	240	320	400	480	560	640	720	800	SO_3	39
40	SO_4	96	192	288	384	480	576	672	768	864	960	SO_4	40
41	Sb	120	240	360	480	600	720	840	960	1080	1200	Sb	41
42	Zn	65.3	130.6	195.9	261.2	326.5	391.8	457.1	522.4	587.7	653.0	Zn	42
43	H	1	2	3	4	5	6	7	8	9	10	H	43

TABLE OF THERMOMETRIC EQUIVALENTS.

According to the Centigrade and Fahrenheit Scales.

C.°	F.°	C.°	F.°	C.°	F.°	C.°	F.°
—40	—40	1	33.8	47	116.6	93	199.4
—39	—38.2	2	35.6	48	118.4	94	201.2
—38	—36.4	3	37.4	49	120.2	95	203
—37	—34.6	4	39.2	50	122	96	204.8
—36	—32.8	5	41	51	123.8	97	206.6
—35	—31	6	42.8	52	125.6	98	208.4
—34	—29.2	7	44.6	53	127.4	99	210.2
—33	—27.4	8	46.4	54	129.2	100	212
—32	—25.6	9	48.2	55	131	101	213.8
—31	—23.8	10	50]	56	132.8	102	215.6
—30	—22	11	51.8	57	134.6	103	217.4
—29	—20.2	12	53.6	58	136.4	104	219.2
—28	—18.4	13	55.4	59	138.2	105	221
—27	—16.6	14	57.2	60	140	110	230
—26	—14.8	15	59	61	141.8	115	239
—25	—13	16	60.8	62	143.6	120	248
—24	—11.2	17	62.6	63	145.4	125	257
—23	—9.4	18	64.4	64	147.2	130	266
—22	—7.6	19	66.2	65	149	135	275
—21	—5.8	20	68	66	150.8	140	284
—20	—4	21	69.8	67	152.6	145	293
—19	—2.2	22	71.6	68	154.4	150	302
—18	—0.4	23	73.4	69	156.2	155	311
—17	1.4	24	75.2	70	158	160	320
—16	3.2	25	77	71	159.8	165	329
—15	5	26	78.8	72	161.6	170	338
—14	6.8	27	80.6	73	163.4	175	347
—13	8.6	28	82.4	74	165.2	180	356
—12	10.4	29	84.2	75	167	185	365
—11	12.2	30	86	76	168.8	190	374
—10	14	31	87.8	77	170.6	195	383
— 9	15.8	32	89.6	78	172.4	200	392
— 8	17.6	33	91.4	79	174.2	210	410
— 7	19.4	34	93.2	80	176	220	428
— 6	21.2	35	95	81	177.8	230	446
— 5	23	36	96.8	82	179.6	240	464
— 4	24.8	37	98.6	83	181.4	250	482
— 3	26.6	38	100.4	84	183.2	260	500
— 2	28.4	39	102.2	85	185	270	518
— 1	30.2	40	104	86	186.8	280	536
— 0	32	41	105.8	87	188.6	290	554
		42	107.6	88	190.4	300	572
		43	109.4	89	192.2	310	590
		44	111.2	90	194	320	608
		45	113	91	195.8	330	626
		46	114.8	92	197.6		

ACETIC ACID.

According to Oudemans.

Percent of absolute Acetic Acid.	Specific Gravity at 15° C.	Percent of absolute Acetic Acid.	Specific Gravity at 15° C.	Percent of absolute Acetic Acid.	Specific Gravity at 15° C.
1	1.0007	34	1.0459	67	1.0721
2	1.0022	35	1.0470	68	1.0725
3	1.0037	36	1.0481	69	1.0729
4	1.0052	37	1.0492	70	1.0733
5	1.0067	38	1.0502	71	1.0737
6	1.0083	39	1.0513	72	1.0740
7	1.0098	40	1.0523	73	1.0742
8	1.0113	41	1.0533	74	1.0744
9	1.0127	42	1.0543	75	1.0746
10	1.0142	43	1.0552	76	1.0747
11	1.0157	44	1.0562	77	1.0748
12	1.0171	45	1.0571	78	1.0748
13	1.0185	46	1.0580	79	1.0748
14	1.0200	47	1.0589	80	1.0748
15	1.0214	48	1.0598	81	1.0747
16	1.0228	49	1.0607	82	1.0746
17	1.0242	50	1.0615	83	1.0744
18	1.0256	51	1.0623	84	1.0742
19	1.0270	52	1.0631	85	1.0739
20	1.0284	53	1.0638	86	1.0736
21	1.0298	54	1.0646	87	1.0731
22	1.0311	55	1.0653	88	1.0726
23	1.0324	56	1.0660	89	1.0720
24	1.0337	57	1.0666	90	1.0713
25	1.0350	58	1.0673	91	1.0705
26	1.0363	59	1.0679	92	1.0696
27	1.0375	60	1.0685	93	1.0686
28	1.0388	61	1.0691	94	1.0674
29	1.0400	62	1.0697	95	1.0660
30	1.0412	63	1.0702	96	1.0644
31	1.0424	64	1.0707	97	1.0625
32	1.0436	65	1.0712	98	1.0604
33	1.0447	66	1.0717	99	1.0580

HYDROBROMIC ACID.

According to Biel.

Percent Br.	Specific Gravity at 15° C.	Percent HBr.	Specific Gravity at 15° C.	Percent HBr.	Specific Gravity at 15° C.	Percent HBr.	Specific Gravity at 15° C.
1	1.0082	14	1.110	27	1.229	40	1.875
2	1.0155	15	1.119	28	1.239	41	1.888
3	1.0230	16	1.127	29	1.249	42	1.401
4	1.0305	17	1.136	30	1.260	43	1.415
5	1.038	18	1.145	31	1.270	44	1.429
6	1.046	19	1.154	32	1.281	45	1.444
7	1.053	20	1.163	33	1.292	46	1.459
8	1.061	21	1.172	34	1.303	47	1.474
9	1.069	22	1.181	35	1.314	48	1.490
10	1.077	23	1.190	36	1.326	49	1.496
11	1.085	24	1.200	37	1.338	50	1.518
12	1.093	25	1.209	38	1.350		
13	1.102	26	1.219	39	1.362		

HYDROCHLORIC ACID.

According to G. Lunge and L. Marchlewski.

Specific Gravity at 15° (H_2O at 4°=1.)	Per Cent. of HCl.	Grams of HCl in 1 Liter.	Specific Gravity at 15° (H_2O at 4°=1.)	Per Cent. of HCl.	Grams of HCl in 1 Liter.
1.000	0.16	1.6	1.100	20.01	220
1.005	1.15	12	1.105	20.97	232
1.010	2.14	22	1.110	21.92	243
1.015	3.12	32	1.115	22.86	255
1.020	4.13	42	1.120	23.82	267
1.025	5.15	53	1.125	24.78	278
1.030	6.15	64	1.130	25.75	291
1.035	7.15	74	1.135	26.70	303
1.040	8.16	85	1.140	27.66	315
1.045	9.16	96	1.145	28.61	328
1.050	10.17	107	1.150	29.57	340
1.055	11.18	118	1.155	30.55	353
1.060	12.19	129	1.160	31.52	366
1.065	13.19	141	1.165	32.49	379
1.070	14.17	152	1.170	33.46	392
1.075	15.16	163	1.175	34.42	404
1.080	16.15	174	1.180	35.39	418
1.085	17.13	186	1.185	36.31	430
1.090	18.11	197	1.190	37.23	443
1.095	19.06	209	1.195	38.16	456
			1.200	39.11	469

NITRIC ACID.

According to G. Lunge and H. Rey.

Specific Gravity at 15° (H_2O at 4°=1.)	Per Cent. of HNO_3.	Grams of HNO_3 in 1 Liter.	Specific Gravity at 15° (H_2O at 4°=1.)	Per Cent. of HNO_3.	Grams of HNO_3 in 1 Liter.
1.000	0.10	1	1.255	40.58	509
1.005	1.00	10	1.260	41.84	521
1.010	1.90	19	1.265	42.10	533
1.015	2.80	28	1.270	42.87	544
1.020	3.70	38	1.275	43.64	556
1.025	4.60	47	1.280	44.41	568
1.030	5.50	57	1.285	45.18	581
1.035	6.38	66	1.290	45.95	593
1.040	7.26	75	1.295	46.72	608
1.045	8.13	85	1.300	47.49	617
1.050	8.99	94	1.305	48.26	630
1.055	9.84	104	1.310	49.07	643
1.060	10.68	113	1.315	49.89	656
1.065	11.51	123	1.320	50.71	669
1.070	12.33	132	1.325	51.53	683
1.075	13.15	141	1.330	52.37	697
1.080	13.95	151	1.335	53.22	710
1.085	14.74	160	1.340	54.07	725
1.090	15.53	169	1.345	54.93	739
1.095	16.32	179	1.350	55.79	753
1.100	17.11	188	1.355	56.66	768
1.105	17.89	198	1.360	57.57	783
1.110	18.69	207	1.365	58.48	798
1.115	19.45	217	1.370	59.39	814
1.120	20.23	227	1.375	60.30	829
1.125	21.00	236	1.380	61.27	846
1.130	21.77	246	1.385	62.24	862
1.135	22.54	256	1.390	63.23	879
1.140	23.31	266	1.395	64.25	896
1.145	24.08	276	1.400	65.30	914
1.150	24.84	286	1.405	66.40	933
1.155	25.60	296	1.410	67.50	952
1.160	26.36	306	1.415	68.63	971
1.165	27.12	316	1.420	69.80	991
1.170	27.88	326	1.425	70.98	1011
1.175	28.63	336	1.430	72.17	1032
1.180	29.38	347	1.435	73.39	1053
1.185	30.13	357	1.440	74.68	1075
1.190	30.88	367	1.445	75.98	1098
1.195	31.62	378	1.450	77.28	1121
1.200	32.36	388	1.455	78.60	1144
1.205	33.09	399	1.460	79.98	1168
1.210	33.82	409	1.465	81.42	1193
1.215	34.55	420	1.470	82.90	1219
1.220	35.28	430	1.475	84.45	1246
1.225	36.03	441	1.480	86.05	1274
1.230	36.78	452	1.485	87.70	1302
1.235	37.53	463	1.490	89.60	1335
1.240	38.29	475	1.495	91.60	1369
1.245	39.05	486	1.500	94 09	1411
1.250	39.82	498	1.501	94.60	1420

NITRIC ACID—Continued.

Specific Gravity at 15° (H_2O at 4° = 1)	Per Cent. of HNO_3.	Grams of HNO_3 in 1 Liter.	Specific Gravity at 15° (H_2O at 4° — 1.)	Per Cent. of HNO_3.	Grams of HNO_3 in 1 Liter.
1.502	95.08	1428	1.512	98.53	1490
1.503	95.55	1436	1.513	98.73	1494
1.504	96.00	1444	1.514	98.90	1497
1.505	96.39	1451	1.515	99.07	1501
1.506	96.76	1457	1.516	99.21	1504
1.507	97.13	1464	1.517	99.34	1507
1.508	97.50	1470	1.518	99.46	1510
1.509	97.84	1476	1.519	99.57	1512
1.510	98.10	1480	1.520	99.67	1515
1.511	98.32	1486			

PHOSPHORIC ACID.

According to A. B. Lyons.★

Specific Gravity at 15° (H_2O at 4°—1.)	Percentage of H_3PO_4.	Specific Gravity at 15° (H_2O at 15°—1.)	Percentage of H_3PO_4.	Specific Gravity at 15° (H_2O at 4°—1.)	Percentage of H_3PO_4.
1.0000	0	1.1816	29	1.4215	58
1.0056	1	1.1889	30	1.4312	59
1.0113	2	1.1962	31	1.4409	60
1.0170	3	1.2035	32	1.4508	61
1.0226	4	1.2110	33	1.4607	62
1.0283	5	1.2184	34	1.4706	63
1.0340	6	1.2260	35	1.4807	64
1.0398	7	1.2336	36	1.4908	65
1.0457	8	1.2412	37	1.5010	66
1.0517	9	1.2489	38	1.5113	67
1.0577	10	1.2567	39	1.5216	68
1.0637	11	1.2645	40	1.5321	69
1.0698	12	1.2724	41	1.5426	70
1.0759	13	1.2804	42	1.5532	71
1.0821	14	1.2885	43	1.5638	72
1.0882	15	1.2967	44	1.5746	73
1.0945	16	1.3050	45	1.5854	74
1.1008	17	1.3134	46	1.5963	75
1.1072	18	1.3219	47	1.6073	76
1.1136	19	1.3304	48	1.6183	77
1.1201	20	1.3391	49	1.6304	78
1.1266	21	1.3479	50	1.6416	79
1.1332	22	1.3568	51	1.6529	80
1.1399	23	1.3657	52	1.6642	81
1.1467	24	1.3748	53	1.6756	82
1.1535	25	1.3840	54	1.6871	83
1.1604	26	1.3932	55	1.6986	84
1.1674	27	1.4026	56	1.7102	85
1.1745	28	1.4120	57		

★ From the Pharmacopœia of the United States.

SULPHURIC ACID.

According to G. Lunge and M. Isler.

Specific Gravity at 15° (H_2O at 4°=1.)	Percent of H_2SO_4.	Grams of H_2SO_4 in 1 Liter.	Specific Gravity at 15° (H_2O at 4°=1.)	Percent of H_2SO_4.	Grams of H_2SO_4 in 1 Liter.
1.000	0.09	1	1.260	34.57	435
1.005	0.83	8	1.265	35.14	444
1.010	1.57	16	1.270	35.70	454
1.015	2.30	23	1.275	36.29	462
1.020	3.08	31	1.280	36.87	472
1.025	3.76	39	1.285	37.45	481
1.030	4.49	46	1.290	38.03	490
1.035	5.23	54	1.295	38.61	500
1.040	5.96	62	1.300	39.19	510
1.045	6.67	71	1.305	39.77	519
1.050	7.57	77	1.310	40.35	529
1.055	8.07	85	1.315	40.93	538
1.060	8.77	93	1.320	41.50	548
1.065	9.47	102	1.325	42.08	557
1.070	10.19	109	1.330	42.66	567
1.075	10.90	117	1.335	43.20	577
1.080	11.60	125	1.340	43.74	586
1.085	12.30	133	1.345	44.28	596
1.090	12.99	142	1.350	44.82	605
1.095	13.67	150	1.355	45.35	614
1.100	14.35	158	1.360	45.88	624
1.105	15.03	166	1.365	46.41	633
1.110	15.71	175	1.370	46.94	643
1.115	16.36	183	1.375	47.47	653
1.120	17.01	191	1.380	48.00	662
1.125	17.66	199	1.385	48.53	672
1.130	18.31	207	1.390	49.06	682
1.135	18.96	215	1.395	49.59	692
1.140	19.61	223	1.400	50.11	702
1.145	20.26	231	1.405	50.63	711
1.150	20.91	239	1.410	51.15	721
1.155	21.55	248	1.415	51.66	730
1.160	22.19	257	1.420	52.15	740
1.165	22.83	266	1.425	52.63	750
1.170	23.47	275	1.430	53.11	759
1.175	24.12	283	1.435	53.59	769
1.180	24.76	292	1.440	54.07	779
1.185	25.40	301	1 445	54.55	789
1.190	26.04	310	1.450	55.03	798
1.195	26.68	319	1.455	55.50	808
1.200	27.32	328	1.460	55.97	817
1.205	27.95	337	1.465	56.48	827
1.210	28.58	346	1.470	56.90	837
1.215	29.21	355	1.475	57.37	846
1.220	29.84	364	1.480	57.83	856
1.225	30.48	373	1.485	58.28	865
1.230	31.11	382	1.490	58.74	876
1.235	31.70	391	1.495	59.22	885
1.240	32.28	400	1.500	59.70	896
1.245	32.86	409	1.505	60.18	906
1.250	33.43	418	1.510	60.65	916
1.255	34.00	426	1.515	61.12	926

SULPHURIC ACID.—Continued.

Specific Gravity at 15° (H_2O at 4°=1.)	Percent of H_2SO_4.	Grams of H_2SO_4 in 1 Liter.	Specific Gravity at 15° (H_2O at 4°=1.)	Percent of H_2SO_4.	Grams of H_2SO_4 in 1 Liter.
1.520	61.59	936	1.745	81.12	1416
1.525	62.06	946	1.750	81.56	1427
1.530	62.53	957	1.755	82.00	1439
1.535	63.00	967	1.760	82.44	1451
1.540	63.43	977	1.765	82.88	1463
1.545	63.85	987	1.770	83.32	1475
1.550	64.26	996	1.775	83.90	1489
1.555	64.67	1006	1.780	84.50	1504
1.560	65.01	1015	1.785	85.10	1519
1.565	65.49	1025	1.790	85.70	1534
1.570	65.90	1035	1.795	86.30	1549
1.575	66.30	1044	1.800	86.90	1564
1.580	66.71	1054	1.805	87.60	1581
1.585	67.13	1064	1.810	88.30	1598
1.590	67.59	1075	1.815	89.05	1621
1.595	68.05	1085	1.820	90.05	1639
1.600	68.51	1096	1.821	90.20	1643
1.605	68.97	1107	1.822	90.40	1647
1.610	69.43	1118	1.823	90.60	1651
1.615	69.89	1128	1.824	90.80	1656
1.620	70.32	1139	1.825	91.00	1661
1.625	70.74	1150	1.826	91.25	1666
1.630	71.16	1160	1.827	91.50	1671
1.635	71.57	1170	1.828	91.70	1676
1.640	71.99	1181	1.829	91.90	1681
1.645	72.40	1192	1.830	92.10	1685
1.650	72.82	1202	1.831	92.30	1690
1.655	73.23	1213	1.832	92.52	1695
1.660	73.64	1222	1.833	92.75	1700
1.665	74.07	1233	1.834	93.05	1706
1.670	74.51	1244	1.835	93.43	1713
1.675	74.97	1256	1.836	93.80	1722
1.680	75.42	1267	1.837	94.20	1730
1.685	75.86	1278	1.838	94.60	1739
1.690	76.30	1289	1.839	95.00	1748
1.695	76.73	1301	1.840	95.60	1759
1.700	77.17	1312	1.8405	95.95	1765
1.705	77.60	1323	1.8410	97.00	1786
1.710	78.04	1334	1.8415	97.70	1799
1.715	78.48	1346	1.8410	98.20	1808
1.720	78.92	1357	1.8405	98.70	1816
1.725	79.36	1369	1.8400	99.20	1825
1.730	79.80	1381	1.8395	99.45	1830
1.735	80.24	1392	1.8390	99.70	1834
1.740	80.68	1404	1.8385	99.95	1838

AMMONIA SOLUTION.

According to G. Lunge and T. Wiernik.

Specific Gravity at 15° (H_2O at 4°=1.)	Percent. of H_3N.	Grams of H_3N in 1 Liter.	Specific Gravity at 15° (H_2O at 4°=1.)	Percent. of H_3N.	Grams of H_3N in 1 Liter.
1.000	0.00	0.0	0.940	15.68	146.9
0.998	0.45	4.5	0.938	16.22	152.1
0.996	0.91	9.1	0.936	16.82	157.4
0.994	1.37	13.6	0.934	17.42	162.7
0.992	1.84	18.2	0.932	18.03	168.1
0.990	2.31	22.9	0.930	18.64	173.4
0.988	2.80	27.7	0.928	19.25	178.6
0.986	3.30	32.5	0.926	19.87	184.2
0.984	3.80	37.4	0.924	20.49	189.3
0.982	4.30	42.2	0.922	21.12	194.7
0.980	4.80	47.0	0.920	21.75	200.1
0.978	5.30	51.8	0.918	22.39	205.6
0.976	5.80	56.6	0.916	23.03	210.9
0.974	6.30	61.4	0.914	23.68	216.3
0.972	6.80	66.1	0.912	24.33	221.9
0.970	7.31	70.9	0.910	24.99	227.4
0.968	7.82	75.7	0.908	25.65	232.9
0.966	8.33	80.5	0.906	26.31	238.3
0.964	8.84	85.2	0.904	26.98	243.9
0.962	9.35	89.9	0.902	27.65	249.4
0.960	9.91	95.1	0.900	28.33	255.0
0.958	10.47	100.3	0.898	29.01	260.5
0.956	11.03	105.4	0.896	29.69	266.0
0.954	11.60	110.7	0.894	30.37	271.5
0.952	12.17	115.9	0.892	31.05	277.0
0.950	12.74	121.0	0.890	31.75	282.6
0.948	13.31	126.2	0.888	32.50	288.6
0.946	13.88	131.3	0.886	33.25	294.6
0.944	14.46	136.5	0.884	34.10	301.4
0.942	15.04	141.7	0.882	34.95	308.3

SOLUTION OF POTASSIUM HYDROXIDE.

According to Gerlach.

Specific Gravity at 15° C.	Percent of KOH.	Specific Gravity at 15° C.	Percent of KOH.	Specific Gravity at 15° C.	Percent of KOH.
1.009	1	1.230	25	1.527	49
1.017	2	1.241	26	1.539	50
1.025	3	1.252	27	1.552	51
1.033	4	1.264	28	1.565	52
1.041	5	1.278	29	1.578	53
1.049	6	1.288	30	1.590	54
1.058	7	1.300	31	1.604	55
1.065	8	1.311	32	1.618	56
1.074	9	1.324	33	1.630	57
1.083	10	1.336	34	1.641	58
1.092	11	1.349	35	1.655	59
1.101	12	1.361	36	1.667	60
1.111	13	1.374	37	1.682	61
1.119	14	1.387	38	1.695	62
1.128	15	1.400	39	1.705	63
1.137	16	1.411	40	1.718	64
1.146	17	1.425	41	1.729	65
1.155	18	1.438	42	1.740	66
1.166	19	1.450	43	1.751	67
1.177	20	1.462	44	1.768	68
1.188	21	1.475	45	1.780	69
1.198	22	1.488	46	1.790	70
1.209	23	1.499	47		
1.220	24	1.511	48		

SOLUTION OF SODIUM HYDROXIDE.

According to Gerlach.

Specific Gravity at 15° C.	Percent of NaOH.	Specific Gravity at 15° C.	Percent of NaOH.	Specific Gravity at 15° C.	Percent of NaOH.
1.012	1	1.279	25	1.529	49
1.023	2	1.290	26	1.540	50
1.035	3	1.300	27	1.550	51
1.046	4	1.310	28	1.560	52
1.059	5	1.321	29	1.570	53
1.070	6	1.332	30	1.580	54
1.081	7	1.343	31	1.591	55
1.092	8	1.353	32	1.601	56
1.103	9	1.363	33	1.611	57
1.115	10	1.374	34	1.622	58
1.126	11	1.384	35	1.633	59
1.137	12	1.395	36	1.643	60
1.148	13	1.405	37	1.654	61
1.159	14	1.415	38	1.664	62
1.170	15	1.426	39	1.674	63
1.181	16	1.437	40	1.684	64
1.192	17	1.447	41	1.695	65
1.202	18	1.456	42	1.705	66
1.213	19	1.468	43	1.715	67
1.225	20	1.478	44	1.726	68
1.236	21	1.488	45	1.737	69
1.247	22	1.499	46	1.748	70
1.258	23	1.508	47		
1.269	24	1.519	48		

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